

CRYOGENIC ADHESIVES

AND SEALANTS—

ABSTRACTED PUBLICATIONS

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PREFACE

This special publication is part of a cryogenic fluids safety review performed by the NASA Lewis Research Center. Major emphasis has been on oxygen safety. The objectives of the review include:

1. Recommendations to improve NASA cryogenics and oxygen handling practices by comparing NASA and contractor systems including the design, inspection, operation, maintenance, and emergency procedures.
2. Assessment of the vulnerability to failure of cryogenic and oxygen equipment from a variety of sources so that hazards may be defined and remedial measures formulated.
3. Formulation of criteria and standards on all aspects of handling, storage, and disposal of oxygen and cryogenic fluids.

This Special Publication is composed of information from the available reports and publications on Cryogenic Adhesives and Sealants. The documents reviewed and abstracted contain experimental data or reviews of the properties of adhesives and sealants at cryogenic temperatures.

These properties include structural and thermoelastic properties, bonding, leak tightness, thermal and physical properties and compatibility properties with oxygen and other cryogenic fluids. The abstracts point out such problems or their solutions where the information is available.

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INTRODUCTION

This survey is made up of information from the available reports and publications on Cryogenic Adhesives and sealants. The documents, listed alphabetically by first author, are original publications of experimental data on properties of adhesives and sealants at cryogenic temperatures, and review papers on cryogenic adhesives and their applications. An abstract has been prepared for each primary document cited, and the most important references are listed as found in the document. In addition, an author index and subject index are provided for the primary documents.

Also listed alphabetically by author are documents considered secondary in nature and include re-publications or variations of the primary documents, progress reports leading to the final reports included as primary documents, and experimental data on adhesive properties at temperatures between about 130 K and room temperature. The citations of the secondary documents include a comment or descriptive line as to the contents of the document.

Paul M. Ordin of the NASA-Lewis Research Center was the Project Manager for NASA.

Identification of a manufacturer's product in this publication in no way implies a recommendation or endorsement by the National Bureau of Standards or by the National Aeronautics and Space Administration.

PRIMARY DOCUMENTS

DEVELOPMENT OF SEALS AND SEALANTS FOR USE AT CRYOGENIC TEMPERATURES

Akawie, R. I.

Hughes Aircraft Co., Culver City, Calif., Final Summary Rept. P64-56,
National Aeronautics and Space Administration Rept. No. NASA-CR-58382,
Contract No. NAS8-2428 (Jun 1964) 71 pp

The objective of the test program was to develop elastomeric materials which could be used as sealants and seals at cryogenic temperatures. The necessary properties for such materials are flexibility and toughness at cryogenic temperatures, and materials which showed the best promise in previous work were silicones and polyurethanes. The program concentrated on synthesis and testing of new silicones and polyurethanes. A poly(methylbutylvinylsiloxane) formulation was tested for thermal contraction between 273 and 77 K, and compared with the same polymer with silica added as filler. As expected, the thermal contraction was less with silica added, but still higher than that of aluminum. Twenty sealant formulations were tested for flexibility, by a bend test at 77 K of a sample of sealant coated on an aluminum strip. Polymers with silica filler or reinforced with commercial short strand alumina-silica fibers showed cracking and loss of adhesion in the bend test, while polymers reinforced with glass cloth generally passed the bend test and developed cracks only to the top layer of glass cloth. The poly(methylbutylvinylsiloxane) showed the best adhesion to aluminum in the 77 K bend tests. The polymer was also the easiest to prepare, with the most consistent properties between batches. The author stated that the good physical properties of this polymer might not be maintained at temperatures down to 20 K, and further testing at lower temperatures was desirable. Further work on synthesizing other siloxane polymers, and urethane polymers containing siloxy bonds, was also recommended.

Important references:

1. Zelman, I. M., Akawie, R. I. and Bahun, C. J., Hughes Aircraft Company, Report No. P62-29, Contract No. NAS 8-2428 (Jun 1962).
2. Zelman, I. M., Akawie, R. I. and Harvey, H., Hughes Aircraft Company, Report No. P63-57, Contract No. NAS 8-2428 (Jun 1963).

BEHAVIOUR OF EPOXIDE ADHESIVE AT LOW TEMPERATURES

Alekseev, V. D., and Malyshev, G. P.

Russ. Eng. J. 53, No. 1, 41-2 (1973). Transl. of Vestn. Mashinostr., No. 1, 35 (1973)

An adhesive was used to provide a vacuum-tight seal with low thermal and electrical conductivity at liquid helium temperature, between a copper cup and a stainless steel fitting. The adhesive was a filled epoxy-polyamide, the components of which are identified by their Russian designations. Bond strengths were found with brass LS-59-1 and with steel 1Kh18N10T adherends, by measuring butt-tensile strengths at 77 and 293 K. Bond strengths with the two adherends were equal at room temperature, and increased at 77 K by 60% with brass and 200% with steel adherends. Brass-brass bonds were subjected to 100 cycles of thermal shock between 77 and 310 K, resulting in 50% loss of bond strength. The bond between a copper cup and a stainless steel fitting was subjected to 100 cycles of thermal shock between 4 and 293 K, and remained leak-tight. The assembly was used for eight months and underwent repeated cycles between 4 and 293 K without any leakage.

Important references:

1. Kharitonov, N. P., et al., Refractory and heat-resistant coatings. Nauka, L. (1969).
2. Timofeev, V. G. and Alekseev, V. D., Adhesives joints. Izd. MDNTP im. Dzerzhinskogo, M. (1970).
3. Samoilov, B. I., Zhurnal tekhnicheskoi fiziki 22, No. 5 (1952).

INVESTIGATION OF BONDING MATERIALS FOR METAL BONDING AT HIGH AND LOW TEMPERATURES (UNTERSUCHUNGEN VON KLEBSTOFFEN FUER METALLKLEBUNGEN ZUR ANWENDUNG BEI TIEFEN UND HOHEN TEMPERATUREN)

Althof, W. (Deutsche Forschungsanstalt fuer Luft- und Raumfahrt, Brunswick, West Germany. Institut fuer Flugzeugbau)
DFL-Mitteilungen, No. 7, 297-304 (1967)

This is a review of previous work by the author and, to a lesser extent, others on adhesives which are suitable for the application of joining metals in which the bonds will be exposed to temperatures from below 120 K to above 600 K. The results given are for tensile-shear strength or shear modulus of lap-joints with the metals being aluminum alloys (Al-Cu-Mg) or stainless steel (17-7 Ph). A graph is given showing tensile-shear strength from 20 K to 323 K for nylon-epoxy, polyurethane, epoxy-phenol, phenol-polyvinyl, epoxy and epoxy-polyamide. The majority of the graphical data given in the paper are for the epoxy-phenol adhesives. The only other results for cryogenic temperatures reported are tensile-shear strengths after long duration (up to 1000 hours) tempering of joints at 78 K. The samples were tested, after the tempering, at 78 K and 293 K. In both situations there was very little degradation and, in fact, some enhancement was evident in the area of 400 hours. The tests ended at 1000 hours, but the curves seem to be leveling out at that point. Most of the paper deals with tests at higher temperatures (to 673 K) as it is evident that long term exposure to temperatures above about 450 K seriously degrades the strength of the adhesive bond regardless of the temperature at which the samples are re-tested. The paper also gives high temperature results for tests (single temperature and after long term treatment) for some ceramic adhesives. These give acceptable values for strength at high temperatures (to 700 K), but no results are given below 273 K.

Important references:

1. Althof, W., Die Festigkeit von überlappten Metallklebungen bei tiefen und hohen Temperaturen und nach Temperaturwechseln. DLR-FB 65-46 (1965).
2. Kausen, R. C., Materials in Design Engng. 60, 94-98 and 108-112 (1964).

STRENGTH OF ADHESIVE BONDED METALS UNDER LOAD OF SHORT AND LONG DURATION
AT LOW AND HIGH TEMPERATURES (FESTIGKEITSVERHALTEN VON METALLKLEBVERBINDUNGEN
UNTER KURZZEIT- UND LANGZEITBEANSPRUCHUNG BEI TIEFEN UND HOHEN TEMPERATUREN)
Althof, W. (Deutsche Forschungsanstalt fuer Luft- und Raumfahrt, Brunswick,
West Germany)
Metall 20, No. 10, 1042-7 (Oct 1966)

This paper gives results of an experimental program to select adhesives for metal bonding in the temperature range 78 K to 673 K. This is a rather wide range for polymeric adhesives as commercially-available high temperature adhesives are not usually suitable at cryogenic temperatures and vice versa. The tests were tensile-shear tests of lap-joints and consisted of initial high and low temperature screening tests followed by tests at various temperatures which were subsequent to long term tempering at various temperatures. These tests were followed by thermal cycling between 248 and 362 K.

Initial tests were made on epoxy, epoxy-nylon, epoxy-polyamid, phenol-polyvinyl, polyimide and epoxy-phenol. All but the epoxy-phenol were unsuitable at high temperatures and even its strength dropped to 1/3 of its value at 78 K when tested at 673 K. Long duration tempering (up to 1000 hours) at 78 K did not significantly reduce shear strength. Long duration tempering at higher temperatures did, however, seriously degrade the strength. The degradation was directly proportional to both the time duration and temperature. For example 20 hours at 573 K reduced the strength, when tested at room temperature, to nearly zero. Thermal cycling between 48 K and 423 K did not seriously (less than 25%) degrade the strength of bonds. The samples were cycled up to 1000 times. The tests were conducted using either stainless steel or aluminum alloys as the metal.

Important references:

1. Rosato, D. V., Adhesives Age 3, No. 12, 38-41 (1960)

THE USE OF VT - 200 ADHESIVE FOR LOW TEMPERATURE VACUUM JOINTS

Anashkin, O. P., and Keilin, V. E. (Institute of Atomic Energy, Moscow, USSR)

Cryogenics 14, ~~7~~, 406-7 (Jul 1974). Also in Instrum. Exp. Tech. 16, No. 6, Part 1, 1589-90 (Nov-Dec 1973). Transl. of Prib. Tekh. Eksp., No. 5, 246-7 (Sep-Oct 1973)

This note describes the use of a Russian adhesive to make leak-tight joints for application at liquid helium temperatures. The adhesive was an epoxy-silicone glue, identified by its Russian designation. The adherends tested were stainless steel, copper, aluminum, and glass-reinforced plastic laminate. Bonds were tested by ten cycles of immersion in liquid nitrogen or liquid helium followed by immersion in hot water. A successful bond was one which remained vacuum tight after the thermal shocks. Successful bonds were obtained between the glass-reinforced laminate and itself, stainless steel, or aluminum; between copper and itself, stainless steel, or aluminum; and between stainless steel and itself. Bonds which withstood the liquid nitrogen thermal shocks also withstood liquid helium shocks. Several examples of applications of the epoxy-silicone adhesive are given, including the sealing of electrical leads through the vacuum space into the liquid helium container of a cryostat, fabrication of a liquid helium cryostat, and joining tubes of different sizes so that the tubes are electrically insulated from each other and from electrical leads passing through the joint.

Important references:

1. Anashkin, O. P., Prib i Tekh. Eksper. No. 2, 225 (1972).
2. Keilin, V. E., Cryogenics 7, 3 (1967).

SELECTION OF A THERMAL BONDING AGENT FOR TEMPERATURES BELOW 1 K

Anderson, A. C., and Peterson, R. E. (Illinois Univ., Urbana. Dept. of Physics and Materials Research Lab.)
Cryogenics 10, No. 5, 430-3 (Oct 1970)

A few materials have been widely used as thermal bonding agents below 1 K, but the question of what material, in a quantitative sense, constitutes a good adhesive for this temperature region has not been fully explored. This paper reviews the available data on thermal bonds and shows that, in general the contact resistance can be considered as comprised of the Kapitza resistance of the interfaces plus the bulk thermal resistance of the bonding agent. The result is that the adhesive can then be chosen on the basis of the desired mechanical properties.

The materials tested were three commercially-available greases, a commercially-available dispersion of copper powder in a proprietary grease, a proprietary varnish, an epoxy, indium metal and a moisture-cured rubber. The bulk resistance of all materials below 1 K can be fitted to $r_B = 7.5 \times 10^{-7} T^{-3} + 20\%$. Below 0.5 K, the thermal resistance of the bonding agent is of little importance if the layer can be made 10^{-3} cm or less thick.

The author points out that the materials tested are essentially for bonding dielectric materials to each other or to metals and not for metal-metal joints.

Important references:

1. Kreitman, M. M. and Callahan, J. T., Cryogenics 10, 155 (1970).
2. McTaggart, J. H. and Slack, G. A., Cryogenics 9, 384 (1969).
3. Anderson, A. C., Reese, W. and Wheatley, J. C., Rev. Sci. Instrum. 34, 1786 (1963).
4. Suomi, M., Anderson, A. C. and Holmstrom, B., Physica 38, 67 (1968).
5. Kreitman, M. M., Rev. Sci. Instrum. 49, 1562 (1969).
6. Krusis, M., Anderson, A. C. and Holmstrom, B., Phys. Rev. 177, 910 (1969).

PROPERTIES OF MATERIALS - 1, APPLICATION OF ADHESIVES

Ashworth, T., and Rechowicz, M. (Manchester Coll. of Science and Technology, England. Physics Dept.)

Cryogenics 8, No. 6, 361-3 (Dec 1968)

Three materials were tested for their suitability as adhesives at low temperatures, particularly for attaching or anchoring heaters, thermometers, thermocouples, or electrical leads in low-temperature calorimetry. The requirements in these applications include resistance to thermal shock and thermal cycling, fast thermal response time across a film, good electrical insulation, ease of application and maintenance, and good vacuum properties. The adhesives considered were a polyvinyl formal, a varnish, and a filled epoxy. Thermal shock resistance was tested by cementing constantan coils to copper tubes, and plunging the assemblies repeatedly into liquid nitrogen and hot water. The polyvinyl formal developed cracks in this situation, and peeled or deteriorated when used only on a small portion of a flat surface. The other materials showed no trace of damage. Thermal conductivities were measured across copper-copper bonds, and relative thermal response times were found across heater-copper bonds. The filled epoxy had the highest thermal conductivity, but it displayed the longest thermal response time because it could be applied only in relatively thick layers. Boundary thermal resistances were not detectable in tests comparing thermal conductivity of a single thick layer of adhesive to that of a number of thin layers with several adhesive-metal boundaries. When surrounded by metal on both sides, the polyvinyl formal showed no sign of the cracking and peeling problems. A method of fabricating flat coils with comparative ease, using either the polyvinyl formal or varnish adhesive, is described.

Important references:

1. Westrum, E. F., Chow, C., Osborne, D. W. and Flotow, H. E., Cryogenics 7, 43 (1967).

SOME MATERIALS FOR ULTRASONIC TRANSDUCER BONDING AT CRYOGENIC TEMPERATURES
Bateman, T. B. (Bell Telephone Labs., Inc., Murray Hill, N. J.)
J. Acoust. Soc. Am. 41, No. 4, 1011-4 (Apr 1967)

The process of bonding piezoelectric transducers to materials at cryogenic temperatures differs in several respects from structural adhesive bonding. The bond most often is not permanent, so that the transducer can easily be removed for re-use. Many of the adhesives for this application are fluids which freeze to produce a bond at cryogenic temperatures. The bond is required to transmit shear and longitudinal waves, but does not have to sustain structural loads. Thickness of the bond is an important parameter, because thick bonds introduce losses. Light liquids can be applied as very thin films, although they may be more difficult to use. A low melting point can be valuable, because it allows most differential thermal contraction during cooling to take place with no stress, before the bond becomes rigid.

This paper lists a number of materials from the literature which have been used successfully for ultrasonic transducer bonding. The materials listed are an epoxy resin, a silicone release compound, a series of silicone fluids varying in viscosity, di-2-ethylhexylsebacate, 4-methyl 1-pentene, natural gas, and a stopcock grease. Assuming successful bonding, the primary consideration in selection is ease of use. The order of choice becomes the stopcock grease, followed by the lower-viscosity silicone fluids, the di-2-ethylhexylsebacate, the 4-methyl 1-pentene, and finally natural gas. The epoxy resin and the release compound are not rated in the list. The author states that his evaluations are based on personal familiarity, and he has not attempted a comprehensive cataloguing of all bonding materials and their relative merits. This review covers the basic considerations in ultrasonic transducer bonding and the most commonly used materials for such bonding at cryogenic temperatures.

A LAMBDA TIGHT LOW TEMPERATURE CEMENT

Beduz, C., Kollen, A., Mathu, F., and Goldschvartz, J. M. (Technische Hogeschool, Delft, Netherlands. Dept. of Applied Physics)
Cryogenics 14, No. 4, 223-4 (Apr 1974)

This brief note describes the successful use of a two component cement, identified by its commercial designation, to provide a leak-tight seal against superfluid helium. The requirements were for a material providing a leak-tight mechanically-strong seal, with good adherence to glass and metal, with high electrical resistivity, and with the flexibility to withstand differential thermal contraction without cracking. In the example shown, the adhesive bonded Pyrex glass, tungsten, and brass to a Pyrex glass tube. The bonds withstood repeated shock cooling by immersion in liquid nitrogen, and remained leak-tight when tested at 1.5 K in liquid helium, and were still leak-tight in liquid helium after three months aging.

Important references:

1. Netzel, Q. G. and Dillinger, J. R., Rev. Sci. Instr. 32, 855 (1961).
2. Wheatley, J. C., Rev. Sci. Instr. 35, 765 (1964).
3. Kausen, R. C., Mat. Desig. Engin. 60, 108 (1964).
4. Anderson, A. C., Rev. Sci. Instr. 41, 1889 (1970).
5. Mota, A. C., Rev. Sci. Instr. 42, 1541 (1971).

DEVELOPMENT OF POTTING COMPOUND FOR CRYOGENIC APPLICATIONS

Boot, R. J., Murphy, C. B., and Smith, O. L. (General Electric Co., Schenectady, N.Y.)

Plast. Des. Process. 2, No. 10, 28-31 (Oct 1962)

The objective of this program was to develop a potting compound with improved compatibility with liquid oxygen. Polyurethanes had been found suitable for casting, but impact sensitive in liquid oxygen. The LOX compatibility was to be improved by the addition of powdered polyfluorocarbon to the polyurethane. Two polyurethane formulations, one without additive and the other containing 46% powdered polyfluorocarbon, were compared in tests of curing characteristics, dielectric strength, coefficient of expansion, sealing properties, adhesive bond strength, and LOX impact sensitivity. Tests were at room temperature except for sealing properties (200 to 365 K), coefficient of expansion (200 to 365 K), and impact sensitivity. The addition of the fluorocarbon had little effect on curing properties and on sealing characteristics, decreased the coefficient of linear expansion between 200 and 365 K, was detrimental to the dielectric strength, and improved the room-temperature aluminum-aluminum adhesive bond strength. Addition of the fluorocarbon also improved the LOX compatibility, showing no impact sensitivity in thicknesses greater than 2 mm. Although not specifically intended for the purposes, the new formulation showed potentiality as a LOX-compatible cryogenic sealant or adhesive.

SPACE ENVIRONMENTAL EFFECTS OF MATERIALS AND COMPONENTS. VOLUME I.
ELASTOMERIC AND PLASTIC MATERIALS AND APPENDIX A THROUGH I
Broadway, N. J., King, R. W., and Palinchak, S. (Battelle Memorial
Inst., Columbus, Ohio)

Army Missile Command, Redstone Arsenal, Ala. Redstone Scientific Information
Center Rept. No. RSIC-150, Contract No. DA-01-021-AMC-203(Z) (Apr 1964)
537 pp

This review is the result of a literature survey on the effects of space environment on elastomeric and plastic materials. The survey covered the effects of combinations of radiation, vacuum, and temperature extremes. Most of the section on structural adhesives is concerned with nuclear or ultraviolet radiation, vacuum, and high temperatures, but one part of the section reviews the combined effects of nuclear radiation and cryogenic temperatures on adhesives. The data reviewed are from Gray, et al. (epoxy, epoxy-phenolic, vinyl-phenolic, nitrile-phenolic, and glass-supported epoxy film adhesives, irradiated in vacuum at RT and tested for tensile-shear strengths at 89 K), and from Yasui (three epoxy and a polyurethane adhesive, irradiated in liquid nitrogen, and tested for tensile-shear strength and flatwise tensile strength at 77 K). These data showed little or no effect of the space environment on bond strengths. A separate appendix reviews the data of Smith (a polyurethane and an epoxy-polyamide adhesive, irradiated and tested for tensile-shear strengths at 20 K, 77 K, and RT) which showed that combined radiation and low temperature increased the strength of the polyurethane and decreased the strength of the epoxy-polyamide.

Important references:

1. Gray, P. E., Cornelius, G. K., O'Donnell, J. D. and Howard, W. W., Aerojet-General Corporation, Azusa, California, RTD-TDR-63-1050, Final Report, AF 04(611)-744 (Feb 1963).
2. Smith, E. T., General Dynamics/Fort Worth, Fort Worth, Texas, FZK-161-2, Annual Report, November 9, 1961 - November 8, 1962, NAS8-2450 (Jan 5, 1963).
3. Yasui, G., Lockheed Aircraft Corporation, Missiles and Space Company, Sunnyvale, California, NSP-63-35, NAS8-5600 (May 7, 1963).
4. Yasui, G., Lockheed Aircraft Corporation, Lockheed-Georgia Company, Marietta, Georgia, ER-6512, 37-69 (Sep 10, 1963).

DEVELOPMENT OF A LIGHTWEIGHT CRYOGENIC INSULATING SYSTEM

Burkely, R. A., Kariotis, A. H., Yoder, F. D., Apisa, J. N., Kost, P., and Rollins, S. R.

Goodyear Aerospace Corp., Akron, Ohio, Final Rept. No. GER-12269, National Aeronautics and Space Administration Rept. No. NASA-CR-76368, Contract No. NAS8-11761 (May 1966)

Considerable testing of adhesive bonds was conducted as part of a two-year program to develop advanced lightweight panel insulation systems for launch vehicle cryogenic propellant tanks. The final insulation panel consisted of an aluminum foil hot face sheet bonded to a heat-resistant phenolic perforated honeycomb panel, which was bonded to an aluminum foil center face sheet, which was bonded to a polyester honeycomb core, which was bonded to a polyester cold face sheet, and the polyester face of the entire panel was bonded to the aluminum tank wall. Bonding to the hot face and center face sheets was done with an epoxy-phenolic adhesive. Bonding to the cold face sheet used a polyurethane, and most of the testing involved this adhesive.

Tests included flatwise-tensile strengths of polyester film to polyester core bonds at 77 and 297 K, with variations in surface preparation and adhesive application; flatwise-tensile, flatwise-compression, edgewise-compression and panel shear strengths of polyester film-core-film panels at 20, 77, 200, and 297 K; and flatwise-tensile strengths of polyester film to aluminum bonds at 20, 77, and 297 K, with variations in primer cure cycle, adhesive cure cycle, and time between priming and bonding. The best surface preparation used a primer coat of heat-cured polyester adhesive. The polyurethane was then roller-coated onto the surfaces, and cured at about 340 K, although a two-day room temperature could develop sufficient bond strength. Panels of the insulation were tested for vibration resistance, thermal conductivity, and thermal effectiveness under multicycle ground hold conditions. The adhesive bonds remained adequate.

Important references:

1. Burkley, R. A. and Shriver, C. B., Final Report, Centaur Insulation Study Program. GER-11193. Contract NAS3-3238. Goodyear Aerospace Corp., Akron, Ohio (1 Nov 1963).
2. McGrew, J. L., Advances in Cryogenic Engineering, Plenum Press, Inc. (1963).
3. Miller, R. N., Bailey, C. D., Beall, R. T. and Freeman, S. M., Advances in Cryogenic Engineering, Plenum Press, Inc. (1963).

COUPLING AGENTS FOR ADHESIVE SYSTEMS

Cassidy, P. E., Johnson, J. M., and Rolls, G. C. (Tracor, Inc., Austin, Tex.)

Ind. Eng. Chem. Prod. Res. Dev. 11, No. 2, 170-7 (Jun 1972)

Coupling agents used as adhesion promoters have improved total adhesive strength and aging resistance of adhesives. Most of the work with coupling agents has been done with glass surfaces and by pretreating the surface with the agent. This paper gives results of a program to investigate a number of adhesion promoters used as integral components of adhesives applied to metallic surfaces. The adhesives considered were a polyurethane and an epoxy, and the 14 coupling agents included silanes, phosphorus esters, chromium complexes, and cyclic and aliphatic amines. Adherends were mild steel, aluminum, and stainless steel. Tests used to evaluate the systems were tensile-shear and T-peel at 77, 297, and 366 K. Initial evaluation used tensile-shear strengths at 297 K with each adhesive, 1% of each coupling agent added, with each of the adherends. Systems showing any improvement over strengths with no coupling agent added were tested further at 297 K to optimize the amount of coupling agent. The optimized systems were then tested for tensile-shear strength at 77 and 366 K. Four polyurethane-aluminum systems were tested for T-peel strength at 297 K, and the two that showed improved strength were tested at 77 and 366 K.

With the polyurethane, four coupling agents increased tensile-shear strength with aluminum, one with mild steel, and one with stainless steel. The useful agents were silanes and a phosphorus ester. Results were consistent over all temperatures. Two of the silanes increased T-peel strengths with aluminum at 297 and 366 K, but all results at 77 K were very low. With the epoxy adhesive, the agents were less effective, and results were not consistent at different temperatures. The agents effective at room temperature often decreased tensile-shear strengths at other temperatures. The useful, or partially useful, agents included silanes, amines, a chromium complex, and a phosphorus ester. At 77 K, one silane improved tensile-shear strength of the epoxy-aluminum system, two silanes and an amine improved epoxy-mild steel, and epoxy-stainless steel was not improved.

Important references:

1. Cassidy, P. E. and Yager, B. J., J. Macromol. Sci., Rev. Polym. Tech., DI, 1 (1971).
2. Thompson, L. M. and Hill, W. E., NASA TM X-53676, George C. Marshall Space Flight Center, Huntsville, AL (Nov 28, 1967).

LOW TEMPERATURE EPOXY-SEALED PLASTIC WINDOWS

Collins, R. L., Mayer, K., Jr., and Travis, J. C. (Texas Univ., Austin, Tex.)

Rev. Sci. Instrum. 38, No. 3, 446-7 (Mar 1967)

This brief note gives the details of a problem and its solution in sealing plastic windows for use at 4.2 K. A mylar film window was sealed with an epoxy resin to a brass fitting, and exposed to liquid helium on one side and vacuum on the other. Two such windows were used successfully for over a year, but when disassembled and rebuilt, they leaked. The epoxy seal showed the presence of cracks. New batches of epoxy and variations of cure cycle did not solve the problem. Finally it was realized that the brass cover plate had been cleaned and smoothed, and that spiral grooves originally machined on the plate had been removed. Machining of grooves into the plate brought immediate success in sealing with the epoxy. The authors conclude that the grooves are crack stoppers, which trap the epoxy into a series of bands, preventing propagation of local cracks and failure of the seal. It is suggested that machining of cover plates on a lathe ordinarily results in a low-amplitude spiral groove, inadvertently contributing to success of low-temperature seals.

Important references:

1. Moss, T. H., Kellers, C. F. and Bearden, A. J., Rev. Sci. Instr. 34, 1267 (1963).
2. Warschauer, D. M. and Paul, W., Rev. Sci. Instr. 27, 419 (1956).

SPECIFIC HEAT OF GE 7031 VARNISH (4 - 18 K)

Cude, J. L., and Finegold, L. (Colorado Univ., Boulder. Dept. of Physics and Astrophysics)

Cryogenics 11, No. 5, 394-5 (Oct 1971)

The specific heat of a varnish was measured between 4 and 18 K. The varnish is a vinyl-modified phenolic which has been used widely as an insulator and adhesive in low temperature work. Its specific heat must be known when it is used as an adhesive in calorimetry. The measurement was made on a stack of copper plates, each of which had been dip-coated with varnish. Specific heat of the varnish is tabulated from 4.188 to 17.45 K, along with data taken by Phillips (see citation in Secondary Documents) in the range from 1.5 to 4 K. The results from the two sets of data do not agree near 4 K, probably reflecting different methods of sample preparation. The authors state that varnish would probably behave more consistently if it were baked, as the manufacturer recommends, rather than air dried and vacuum dried, as it is often used in cryogenic applications. Despite the discrepancy, the authors conclude that the results are good enough to be useful in correcting for small quantities of varnish used in low temperature calorimetry.

Important references:

1. Wheatley, J. C., Griffing, D. F. and Estle, T. L., Rev. Sci. Instrum. 27, 1070 (1956).
2. Ashworth, T. and Rechowicz, M., Cryogenics 8, 361 (1968).
3. Lowndes, D. H., Finegold, L., Rogers, R. N. and Morosin, B., Phys. Rev. 186, 515 (1969).
4. Phillips, N. E., Phys. Rev. 114, 676 (1959).

GLUING OF LAVSAN TO METAL

Davletshin, R. I., and Radkevich, I. A. (Institute of Theoretical and Experimental Physics, Moscow, USSR)
Instrum. Exp. Tech. 16, No. 4, Pt. 2, 1279-80 (Jul-Aug 1973); Transl. of Prib. Tekh. Eksp. No. 4, 250-1 (Jul-Aug 1973)

An adhesive was formulated for bonding polyethyleneterephthalate film to itself, to metal, or to plexiglass, at temperatures as low as 77 K. The adhesive was made up of an epoxy resin, identified by its Russian designation, mixed with polyethylenepolyamine. Polyethyleneterephthalate film-metal bonds, with the metal not specified, were tested for "non-uniform stripping," a peel strength, at 77 K and RT. The effects of varying the adhesive formulation, curing cycle, and diluents for viscosity control were measured and optimized. The results shown in the paper are at RT, but the text states that cooling to 77 K and thermal cycling between 77 K and RT have no effect on bond strength within the measurement error limits, for thin bonds up to 0.3-0.5 mm thick. Thicker bonds crack with shock cooling. The adhesive was used successfully to fabricate the vacuum housing of a liquid hydrogen target.

Important references:

1. Kardashov, D. A., Synthetic Glues (in Russian), Khimiya (1968).
2. Vasil'ev, L. M., Radkevich, I. A., Sokolovskii, V. V. and Smolyankina, T. G., Pribory i Tekh. Eksperim., No. 4, 56 (1971).

THERMAL CONDUCTIVITY OF ADHESIVES AT LOW TEMPERATURES

Denner, H. (Fritz-Haber-Institut, Max-Planck-Gesellschaft, Berlin-Dahlem, Germany)

Cryogenics 9, No. 4, 282-3 (Aug 1969)

The thermal conductivities of two adhesives were measured. The adhesives were suited for attaching samples, thermoresistors, thermocouples, or heating wires to cooled apparatus at cryogenic temperatures. For such applications, the adhesive is required to combine high thermal conductivity with high electrical resistance. The adhesives investigated were commercial materials, one cement adhesive and one epoxy adhesive. Thermal conductivities of long cylindrical cured samples were measured between 10 and 100 K, and shown graphically, where they fall between the conductivities of metals and of glass and polymers. It is noted that the cement adhesive shrinks during hardening, and must be used only in very thin layers to prevent cracks and pores.

ADHESIVE BONDING FOR METAL-LINED FILAMENT-WOUND CRYOGENIC PRESSURE VESSELS

Doyle, H. M.

Douglas Aircraft Co., Inc., Santa Monica, Calif., Missile and Space Systems Div., Paper No. 3816, Contract No. NAS3-6293 (1966) 21 pp. Presented at the Cryogenic Engineering Conference, Boulder, Colo., June 13-15, 1966

The experimental program was undertaken to select the most suitable of available commercial adhesives for bonding a metallic foil liner to the inside of an epoxy-glass composite filament-wound pressure vessel. The adhesive must maintain bond integrity during repeated cyclic pressure loading of the pressure vessel at temperatures from 20 to 298 K. The adhesives tested included an epoxy, a polyurethane, a nylon-epoxy, and a polyurethane-epoxy blend, each reinforced with either a glass or nylon fabric. Adherends were either aluminum or nickel, representing the metallic liner, and epoxy-glass composite, representing the pressure vessel. The nickel proved difficult to bond, even with chemical etchants and cleaners applied. Good adhesion to nickel was obtained by using a silane primer before applying the adhesive, although the epoxy refused to adhere even with this treatment.

Initial tests were tensile-shear and drum-peel tests of aluminum-composite and nickel-composite bonds at 20, 77, and 298 K. In most cases, polyurethane and polyurethane-epoxy adhesives were superior to the epoxy and nylon-epoxy adhesives at cryogenic temperatures. For all adhesives, peel strengths with nickel were about equal to those with aluminum. For the polyurethane and polyurethane-epoxy adhesives, with nylon scrim the peel strengths with nickel decreased at cryogenic temperatures, while with glass scrim the peel strengths with nickel increased at cryogenic temperatures. These two adhesives with each of the two reinforcements were made into laminated samples for further testing, for ultimate tensile strength, elongation, and elastic modulus at 20, 77, and 298 K, and for thermal contraction from 298 to 198, 77, and 20 K. Higher ultimate elongations were observed with nylon scrim for both adhesives, and with polyurethane-epoxy adhesive for both scrims. Thermal contraction coefficients of the adhesives with glass reinforcement were nearer to those of the composite and metal liner, than were those of the adhesives with nylon reinforcement. The polyurethane-epoxy blend adhesive was chosen for use in fabrication of vessels for further test programs.

Important references:

1. Roseland, L. M., Douglas Paper No. 1666. Presented at the National Aeronautic and Space Engineering and Manufacturing Meeting, Los Angeles, California, Sep 1963.
2. Lantz, R. B., Douglas Paper No. 1750. Presented at the 6th National SAMPE Symposium, Seattle, Washington, Nov 1963.

PERMEATION OF ADHESIVELY BONDED JOINTS BY GASEOUS AND LIQUID HYDROGEN
Egger, C. T., Nauman, D. A., Carter, T. J., and Gayle, J. B.
National Aeronautics and Space Administration, Marshall Space Flight
Center, Huntsville, Ala., Tech. Memo. X-53066 (Jun 1964) 30 pp

There is a requirement for repair procedures in the liquid hydrogen fueled S-II and S-IVB stages of the Saturn V rocket which uses adhesives as sealants. Specifically the adhesives are used to bond the intermediate bulkhead of the tank, and thus come in contact with hydrogen and must not be permeable to either liquid or gaseous hydrogen.

The tests used annular plates of 2014 aluminum alloys with the center hole covered by a circular plate of the same material adhesively bonded to the annular plate. These were then tested for leakage of gaseous hydrogen and helium using a mass spectrometer. Tests were made on eight commercially-available adhesives including heat-cured epoxy, amine cured epoxy, polyurethane and four epoxy-phenolics. Non-filleting adhesives were several orders of magnitude less permeable to hydrogen than the filleting epoxy-phenolics. There was, however, great variability among samples and many bonds were found to be broken and thus had mechanical leaks. The narrower adhesive overlap specimens performed better than the wider ones and there was a slight correlation with bond thickness (thinner performed better).

The liquid phase studies were done to determine which adhesives retained their structural integrity on exposure to liquid hydrogen. The experimental procedure utilized 74 layers of annular circular plates bonded together with the adhesives giving an effective bond length of about 10 meters. These were tested first for hydrogen gas permeability and then for liquid hydrogen permeability. Most of the tested cells failed on exposure to liquid hydrogen with only 4 of 12 surviving without rupturing. Two of the epoxy formulations proved adequate.

The authors concluded that for non-filleting adhesives the determining factor is the ability to survive exposure to liquid hydrogen rather than permeability per se. Attempts to analyze the data in terms of flow mechanisms and physical parameters were unsuccessful.

BONDING PLASTIC TO METAL FOR HIGH STRENGTH AT LOW TEMPERATURES

Eppinger, C. E., and Love, W. J. (Denver Research Inst. Colo.)

Adv. Cryog. Eng. 4 (Proc., Cryogenic Engineering Conference, Cambridge, Mass., September 3-5, 1958), 123-31, K. D. Timmerhaus, Ed., Plenum Press Inc., N.Y. (1960)

Tests of six commercial adhesives were conducted to establish a satisfactory means of attaching a teflon facing to a metal part for cryogenic application. The adhesives considered were identified only by their commercial designations.

SAE 52100 steel was used as the metal adherend. The adhesives were tested for tensile-shear strength of steel-teflon bonds at 77 K. The materials fell into two strength groups with three of the adhesives exhibiting about twice the bond strength of the others. The addition of aluminum dust to one adhesive in amounts up to 40 wt. % filler resulted in no gross differences in strength. AF 13 was selected for further study because of its ease of use, being a one-part film style adhesive requiring no prime coat or other preliminary operation. The further study resulted in optimization of cure cycle, metal surface preparation, and teflon etch. A satisfactory bonding system was devised within the material limits of the adherends. The authors state that further advantage might be gained by selecting adherends with more closely matched thermal expansion coefficients, such as aluminum, bronze, or stainless steel bonded to a facing of graphite-filled teflon laminated with a glass fiber fabric base.

LIQUID HYDROGEN TANK INSULATION TESTS

Evenson, J. H.

General Dynamics/Astronautics, San Diego, Calif., Rept. No. GDA-10E-1483
(Mar 1962) 36 pp

The purpose of the test program was to evaluate a foam-filled honeycomb insulation and the adhesives used to bond it to an aluminum tank, under conditions of cryogenic temperatures and thermal and pressure stresses. Nine adhesives, identified only by their commercial designations, were tested. The insulation was a phenolic resin-glass fiber sine-wave honeycomb filled with polyurethane foam. In the first test, six molded panels of the polyurethane foam were bonded to the outside of an aluminum tube with five different adhesives. The outside of each panel was covered with a glass cloth-epoxy resin protective coating. The assembly was cycled twenty times between 20 K and 260 K or above, by filling the tube with liquid hydrogen, then allowing it to boil off. After cycling, three of the adhesives had debonded and two were intact. The second test used two adhesives to bond the honeycomb insulation to the outside of an aluminum tube. Joints were simulated with inserts of honeycomb, foam, and glass cloth-epoxy wedges, and the outside of the assembly was covered with glass cloth-epoxy coating. The tube was filled with liquid hydrogen, and pressurized 20 times at 20 K. The structure remained intact, except for some delamination of the glass cloth-epoxy outer covering. The third test was of internal insulation. The foam-filled honeycomb insulation was built on a mandrel, then bonded to the inside of the aluminum tube. The inner surface and exposed ends were protected with glass cloth-epoxy layer, and glass cloth-epoxy strip covered each joint. The insulated tube was filled with liquid hydrogen and pressurized 20 times at 20 K. The glass cloth-epoxy strips and end coverings showed debonding, but the bond to the aluminum remained intact. Of the nine adhesives tested, four had bond failures at 20 K, including the epoxy used with the glass cloth for protective layers. Two of the remaining adhesives, both epoxies, were also easy to apply and cure, and were considered effective for bonding the foam-filled honeycomb insulation to aluminum liquid hydrogen tanks, either internally or externally.

THE STRENGTH OF TEN STRUCTURAL ADHESIVES AT TEMPERATURES DOWN TO -424°F
Frost, W. M. (National Bureau of Standards, Boulder, Colo. Cryogenic
Engineering Lab.)
Adv. Cryog. Eng. 5 (Proc., Cryogenic Engineering Conference, Berkeley,
Calif., September 2-4, 1959) 375-84, K. D. Timmerhaus, Ed., Plenum
Press, Inc., N.Y. (1960)

Several types of structural adhesives were evaluated at low temperatures. The materials were commercial adhesives of different combinations of epoxy, vinyl, and phenolic resins, including a rubber-epoxide phenolic, two nitrile rubber-phenolics, four vinyl-phenolics, a filled liquid epoxide, and two epoxy-phenolics supported on glass cloth carriers, one of which was filled with aluminum dust.

The adhesives were tested in aluminum-aluminum lap joints, except for the last two which were tested in stainless steel-stainless steel lap joints. Shear-tensile tests were performed at 196, 76, and 20 K with gradual cooling to the test temperature, and at 76 K with shock cooling to the test temperature. The bond strengths generally decreased at lower temperatures, although highest strengths of the epoxy-phenolic types were observed between 76 and 196 K. At 20 K, the epoxy-phenolic adhesives had the highest bond strengths, followed by the filled epoxy, the vinyl-phenolic, and the rubber-phenolic in order of lower bond strengths. The author concludes that, of the material types tested, the most suitable adhesives for low-temperature use would employ epoxy or epoxy-phenolic resins supported on a glass cloth film and filled with metallic dust to a degree necessary to match the thermal expansion of the adhesive to that of the adherend.

Important references:

1. McClintock, R. M. and Hiza, M. J., Modern Plastics (June 1958).
2. Adhesive Properties, -300 to $+700^{\circ}\text{F}$ (ASTIA Document No. AD151529).

MAGNETIC SUSCEPTIBILITIES OF SOME MATERIALS WHICH MAY BE USED IN CRYOGENIC APPARATUS

Ginsberg, D. M. (Illinois Univ., Urbana. Dept. of Physics and Materials Research Lab.)

Rev. Sci. Instrum. 41, No. 11, 1661-2 (Nov 1970)

The magnetic susceptibilities of twelve materials were measured to determine whether the materials are sufficiently nonmagnetic to be useful in building cryogenic equipment for experiments in which distortion of an applied magnetic field must be avoided. The materials include 5 adhesives, identified by their commercial designations, including epoxies, modified polyurethanes, and a room temperature curing silicone rubber fluid. Measurements were made between 1.17 and 4.20 K. Results are presented in the form of coefficients of an equation relating magnetic susceptibility to density of the material. The author concludes that the results are sufficiently precise to indicate clearly which materials can be used without disturbing an applied magnetic field.

Important references:

1. Salinger, G. L. and Wheatley, J. C., Rev. Sci. Instrum. 32, 872 (1961).
2. Commander, R. J. and Finn, C. B. P., J. Sci. Instrum. (J. Phys. E) 3, 78 (1970).

ZERO LEAKAGE DESIGN FOR DUCTS AND TUBE CONNECTIONS FOR DEEP SPACE TRAVEL.
VOLUME II. CONNECTOR CONCEPT STUDIES

Gitzendanner, L. G.

General Electric Co., Schenectady, N. Y., Final Rept. No. S-67-1157-vol-2, National Aeronautics and Space Administration Rept. No. NASA-CR-93926, Contract No. NAS8-11523 (Aug 1967) 83 pp

Adhesive bonding was investigated as a means of producing permanent or semi-permanent leak-tight connections and joints of tubes and ducts. Tapered joints were used to increase the bond area to make a stronger joint. The adhesive was an epoxy, unfilled for the first two test series and filled with talc for the third series. Adherends were tubes, and the materials were not specified, although aluminum and stainless steel were mentioned in connection with the tests.

In the first test series, samples were made with plain tapered joints with different angles of taper. Four samples were pressure checked in a vacuum chamber at room temperature, and proved to be leak-tight. Samples for the second test series had a machined-in shoulder to adjust different adhesive film thicknesses. Two samples were fatigue tested for 100,000 cycles without failure, and remained leak-tight. Since the plain tapered joints gave better results, the third series used plain tapered joints and filled epoxy adhesive. All but one of the 25 samples were leak-tight. Five samples were pull-tested for tensile strength at room temperature. Eight samples were cycled between 218 and 358 K five times, and three of them were pull-tested at room temperature. Since the thermal cycling had no effect on tensile strength, the other five samples were again thermally cycled, this time between 91 and 358 K five times. Two of these samples were pull-tested at room temperature and three were pull-tested at 91 K. The samples pulled at 91 K had about 50 percent greater strength than those tested at room temperature, even though the test involved one more temperature shock before loading. Eight other samples from the third test series were vibrated under various loads without losing strength or leak-tightness.

The authors conclude that adhesive bonding can be considered for making leak-tight connections wherever compatibility between the adhesive and the working fluid are established.

DEVELOPMENT OF CRYOGENIC ADHESIVES WITH +150 DEGREES C CAPABILITY
Glasgow, D. G., and McClung, C. E. (Monsanto Research Corp., Dayton,
Ohio)

Aerospace Adhesives and Elastomers (Proc. National SAMPE Technical
Conference, 2nd, Dallas, Tex., Oct 6-8, 1970), Vol 2, 33-47, Society of
Aerospace Material and Process Engineers (1971)

The object of the program was to develop an adhesive for aluminum-aluminum bonds having useful tensile-shear and T-peel strengths from 20 to 423 K, with a pot life of two hours or more and easy processing characteristics. The best previous adhesive was a polyurethane which had insufficient tensile-shear strength at 423 K and too short a pot life. Commercially available components were used to formulate new polyurethane adhesives, which were evaluated to determine the effects of chemical structure on properties. The best bond strengths were associated with short pot lives, and several diisocyanates were synthesized in attempts to modify the reaction rates, and lengthen pot life. Initial evaluations of the adhesives tested aluminum-aluminum bonds for tensile-shear strengths at 77, 296, 355, and 406 K and for T-peel strengths at 296 K. Later evaluations used tensile-shear and T-peel tests at 77, 296, and 423 K, and the best adhesives were tested for tensile-shear strengths at 4 K. Pot lives were estimated by measuring the change of relative viscosity with time after the adhesive was formulated. The program resulted in the development of a polyurethane adhesive which satisfied the goals of tensile-shear strength from 4 to 423 K and T-peel strength from 77 to 423 K, had a pot life greater than 2 hours, and could be processed in the same manner as previously-available polyurethanes.

Important references:

1. Smith, M. B. and Susman, S. E., *Advan. Cryogenic Eng.* 8, 300 (1963).
2. Kausen, R. C., *Materials Design Eng.* 60, 108 (Sep 1965).
3. Bodnar, M. J. and Kelly, E. R., *Adhesives Age* 2, 29 (apr 1959).
4. Roseland, L. M., Seventh National SAMPE Symposium, May 1964, Section 7.
5. Sandler, S. R. and Berg, F. R., *J. Appl. Poly. Sci.* 9, 3909 (1965).
6. Hertz, J., *Advan. Cryogenic Eng.* 7, 336 (1962).
7. Sandler, S. R. and Berg, F. R., *J. Appl. Poly. Sci.* 9, 3707 (1965).
8. Sandler, S. R., *J. Appl. Poly. Sci.* 11, 811 (1967).

SYNTHESIS OF FLUORINATED POLYURETHANES

Gosnell, R., and Hollander, J. (Narmco Research and Development, San Diego, Calif.)

Cryogenic Properties of Polymers (Proc., NASA-Case Conference on the Properties of Polymers at Cryogenic Temperatures, Cleveland, Ohio, April 25-27, 1967), 279-98, T. T. Serafini, et al., Eds., Marcel Dekker, Inc., N.Y. (1968). Also in J. Macromol. Sci., Phys. B1, No. 4, 831-50 (1967)

This paper reports results of an investigation to develop structural adhesive systems which are compatible with liquid oxygen. Based upon previous materials work, the study concentrated on the preparation of fluorinated polyurethanes. The adhesive systems are to be used to bond clips, studs, brackets, doubler strips, insulation, etc. in liquid oxygen fueled aerospace vehicles. A further constraint on the systems was the provision for curing under ambient conditions. The first materials studied were highly fluorinated aliphatic diamines, diisocyanates, polyesters and polyethers. Polyurethanes were successfully prepared from polyhexafluoropentamethylene adipate, polyhexafluoropentamethylene malonate, polyhexafluoropentamethylene carbonate, polyhexafluoropentamethylene formal, polytrifluoropropylene oxide and polyperfluoropropylene oxide.

The liquid oxygen compatibility results revealed only three formulations which exhibited no reactions in 20 tests on the ABMA Impact Tester. These three materials are polyhexafluoropentamethylene perfluorotrimethylene dicarbamate, polyhexafluoropentamethylene hexafluoropentamethylene dicarbamate and polyhexafluoropentamethylene tetrafluoro-p-phenylene dicarbamate. Some long term (60 hours) immersion tests were also conducted in nitrogen tetroxide with no adverse effects.

Important references:

1. Holland, D. G., Moore, G. J. and Tamborski, C., Chem. Ind. (London), 1376 (1965).
2. Yakobson, G. G., et al., Zh. Obschch. Khim. 134, No. 10, 3514 (1964).

SOME EFFECTS OF STRUCTURE ON A POLYMER'S PERFORMANCE AS A CRYOGENIC ADHESIVE

Gosnell, R. B., and Levine, H. H. (Whittaker Corp., San Diego, Calif.) *Polymers in Space Research* (Proc., American Chemical Society Symposium, Pasadena, Calif., July 15-17, 1968), 345-57, C. L. Segal, et al., Eds., Marcel Dekker, Inc., N. Y. (1970). Also in *J. Macromol. Sci., Chem.* A3, No. 7, 1381-93 (Nov 1969)

This paper summarizes an extended investigation into the development of polymeric adhesives which exhibit high strength from 20 K to above 600 K. The authors have developed a new approach which consists of designing polymers with segmented chain motion which is not frozen out below the glass transition temperature. This has been shown to be feasible in polymers with quite high glass transition temperatures (around 700 K). Lap shear tensile test results are shown for a number of adhesives at 77 K to show their relative merits. The adhesives include nylon epoxy, epoxy polyamide, epoxy, epoxy phenolic, rubber phenolic, polyurethane, heterocyclic aromatic and vinyl phenolic. Polyurethane adhesives have vastly higher strength at 77 K. The three types of polyurethanes studied were based on polyester (poor performance), propylene oxide (acceptable performance), and tetrahydrofuran polyether (outstanding performance). The authors feel the performance of the latter is a result of the availability of rotational freedom far below the glass transition temperature. T-peel test results are shown for this adhesive from 20 K to 370 K.

The good results shown with the above led to studies of polybenzimidazoles (PBI). Results, to 20 K, on PBI are shown for tensile-shear, flexural strength, modulus and torsional braid analysis tests. In all, good performance is exhibited. Impact tests on bulk PBI at 77 K show no degradation from room temperature values. PBI shows no signs of embrittlement in these tests even though they were done more than 300 K below the glass transition temperature. The authors also report that polyimides and polyquinoxalines exhibit similarly outstanding performance at 77 K.

Important references:

1. Gillham, J. K., *Polymer Preprints* 7, 513 (1966).

ROCKETS IN SPACE ENVIRONMENT. VOLUME 1 - THE EXPERIMENTAL PROGRAM
Gray, P. D., Cornelius, G. K., O'Donnell, J. D., and Howard, W. W.
Aerojet-General Corp., Azusa, Calif., Final Rept. No. 2484, Air Force
Systems Command, Edwards AFB, Calif., Rept. No. RTD-TDR-63-1050, Contract
No. AF-04(611)-7441 (Feb 1963) 260 pp

A program was carried out to establish design criteria for space propulsion systems. Part of the program consisted of determining the behavior of rocket engine materials and components in the space environment. Among the materials tested, a number of resins were considered for composites with glass cloth. Lap-shear specimens were made with some of the resins, and in this case the resins act as adhesives. The resins tested were an epoxy, an epoxy-phenolic, a vinyl-phenolic, a nitrile phenolic, and a glass-supported epoxy film. The adherends were not specified. Tensile-shear strengths at 89 K were obtained for control samples, and for samples exposed to vacuum, to gamma radiation, and to both vacuum and radiation. Environmental exposures had no apparent effect on tensile-shear strengths, with variations between treatments being about as large as the random scatter of data caused by fabrication and testing techniques. The glass-supported epoxy film had the highest tensile-shear strength, followed in order by the epoxy, vinyl-phenolic, nitrile-phenolic, and epoxy-phenolic. Since the strengths of the resins were lower than those of resin-filler composite systems, the authors conclude that the strength of a composite is more dependent on the strength of the filler material than on the strength of the resin.

IGNITION IN HIGH PRESSURE OXYGEN

Guter, M.

British Oxygen Co., Ltd., London, England, R. and D. Rept. No. 1312,
Ministry of Aviation S and T Memo 13/50 (Feb 1967) 56 pp

This report gives results of a 1950 study to measure the ignition temperature of a large number of materials in high pressure (to 250 atmospheres) oxygen gas. The classes of materials tested were: 1) lubricants, including thread sealants, 2) natural and synthetic rubber hose material, 3) polymers, 4) valve seat materials and 5) metals and alloys. This abstract covers only 1) above. The thread sealants tested were a British Military anti-seize and sealing material, a commercial thread sealing and lubricating compound, and silicone greases. None of the materials ignited below 450 K at pressures up to 250 atmosphere. The ignition temperature fell with increasing oxygen pressure, but the effect was not marked at pressures above 50 atmospheres up to the maximum of 250 atmospheres. The two thread sealants had slightly higher ignition temperatures than the silicone greases and were thus recommended. The ignition temperatures of the former were around 650 K at 250 atmospheres while that of the latter was about 400 K at the same pressure.

EVALUATION OF ADHESIVES FOR SEALING METALLIC AND PLASTIC FILMS FOR USE AT LIQUID-HYDROGEN TEMPERATURES

Hacker, P. T., DeVos, F. J., and Stutesman, H. L. (National Aeronautics and Space Administration, Lewis Research Center, Cleveland, Ohio)

National Aeronautics and Space Administration Tech. Memo. No. NAS-TM-X-1057 (Feb 1965) 12 pp

The investigation was conducted to evaluate the ability of fifteen different adhesives to provide a hermetic seal in lap joints between thin metallic foils and plastic films in the presence of gaseous and liquid hydrogen. A laminated composite of metallic foil and plastic film is attractive for use as an impermeable inner lining for filament-wound fiberglass liquid hydrogen rocket fuel tanks. To make such an inner lining, the composite must be joined to itself with a hermetically sealed joint. If the liner conforms to the inner tank wall, the joint carries no load, and the most important factor in the adhesive is sealability rather than strength.

The adhesives tested included a polyurethane, four epoxy-polyamines, a nylon-filled epoxy-polyamine, three epoxy-polyamides, an epoxy, two rubber base adhesives, a urethane-polyester, and two polyesters. Adherends were 3-ply laminates of aluminum foil and mylar, some with aluminum outside and some with mylar outside, so that the bonds tested were aluminum-aluminum or mylar-mylar lap joints. A test-sample consisted of two squares of laminate bonded around the perimeter with the test adhesive, and constructed so that the enclosed volume could be evacuated. Test procedure consisted of room temperature leak check, shock cooling to 77 K followed by room temperature leak check, three cycles of shock cooling to 20 K and leak testing at 20 K followed by warming to room temperature, and a final room temperature leak check. Loss of vacuum at any time constituted a failure.

The epoxies and nylon-filled epoxies were the most satisfactory of the adhesives tested, with an overall failure rate of less than 8 percent. The urethane systems had a failure rate of 25 percent. The polyester failure rate was 50 percent with all failures in aluminum-aluminum bonds. The rubber based adhesives had a failure rate of 100 percent before the first cooling to 20 K.

Important references:

1. Hertz, J., Adhesives Age 4, No. 8, 30-37 (Aug 1961).
2. Smith, M. B. and Susman, S. E., Narmco Res. and Dev. (May 1963).
3. Zelman, I. M., Akawie, R. I. and Harvey, H., Summary Rep., Hughes Aircraft Co. (Jun 30, 1963).

DEVELOPMENT OF MODIFIED POLY(PERFLUOROPROPYLENEOXIDE) URETHANE SYSTEMS
FOR USE IN LIQUID OXYGEN AND IN ENRICHED 100 PERCENT OXYGEN ATMOSPHERE
Harrison, E. S.

Whittaker Corp., San Diego, Calif., Research and Development Div. Final
Rept., 22 April 1971 - 23 August 1973, National Aeronautics and Space
Administration Rept. No. NASA-CR-120104, Contract No. NAS8-27087 (Sep
1973) 67 pp

This two-phase program was directed toward improving the mechanical and adhesive properties of the LOX-compatible poly-fluorinated polyurethane resin system, and to investigating the feasibility of using such an adhesive system in a weld-bond configuration for liquid oxygen tankage. In a previous program, a fluorinated adhesive was developed that showed excellent liquid oxygen compatibility and good strength at low temperature, but had deficient strength at temperatures above 340 K. The first phase of the program attempted to improve the properties of the adhesive by using different curing agents. Experimental formulations were tested in aluminum-aluminum bonds for tensile-shear strengths at 77 K, RT, and 367 K. Improvements in bond strengths at 367 K with the new curing agents were minor, and were accompanied by some loss of low-temperature strength.

The second phase of the program was first directed toward improving the reproducibility and reliability of the adhesive properties and oxygen compatibility of the adhesive. Samples of the adhesive tended to show a low level of sensitivity to impact in oxygen, due to contamination introduced in processing. Extreme care in purification and processing kept oxygen impact sensitivity very low, but at the cost of severe loss of adhesive properties. The use of silane-modified epoxy primers improved the adhesive properties, as shown by tensile-shear strengths at 77 K, RT, and 367 K and bell-peel strengths at 77 K and RT of aluminum-aluminum bonds.

The second part of the second phase of the program was concerned with weld-bond joining of aluminum. The joints were made both by weld-through of uncured adhesive and by capillary fill-in of spot-welded adherends. Acceptable bonds were obtained, and use of the primer produced superior results, as shown by tensile-shear strengths at 77 K, RT, and 367 K. When the primer was used, it was necessary to fill the adhesive with aluminum powder to lower electrical resistance enough for welding. Because several samples became badly contaminated, only a few tests for oxygen compatibility were made, but these impact tests on weld-bond aluminum joints in liquid oxygen gave excellent results, showing no failures in forty specimens. The author concludes that high quality bonds are readily attained by both fabrication methods.

SPECIFIC HEAT OF GENERAL ELECTRIC 7031 VARNISH FROM 2 TO 80 K
Heessels, J. T. (Technische Hogeschool, Eindhoven, Netherlands)
Cryogenics 11, No. 6, 483-4 (Dec 1971)

This brief paper reports measurements on the specific heat of a varnish which is often used as an insulating adhesive in low-temperature work. The varnish, a polyvinyl phenolic, was deposited in a thin layer, air dried, and rolled into a cylinder for measurement. The specific heat was measured over the range from 2 to 80 K. The test method allowed an estimate of thermal conductivity of the varnish at 4 and 10 K. Specific heat results are presented graphically, and show an increase nearly proportional to temperature between 20 and 60 K, and nearly proportional to T^3 below 5 K. Comparison of these specific heat and thermal conductivity results with measurements made in other experiments shows systematic differences, and the author concludes that the differences are caused by variations in sample preparation.

Important references:

1. Phillips, N. E., Phys. Rev. 114, 676 (1959).
2. McTaggart, J. H. and Slack, G. A., Cryogenics 9, 384 (1969).

ADHESIVES FOR EXTREME-LOW-TEMPERATURE APPLICATIONS

Hertz, J. (General Dynamics/Astronautics, San Diego, Calif.)

Electro-Technology 70, No. 3, 93-6 (Sep 1962)

This paper summarizes the results of projects designed to test and select adhesives for aerospace applications at cryogenic temperatures. In particular it is directed toward the designer/engineer with a requirement to bond electrical and electronic devices and accessories to structural members which must operate in a cryogenic environment. The data reported (in graphical form) cover shear-tensile, butt-tensile and impact testing as well as qualitative results of vibration tests. Test results are shown at 20, 77, 200 and 310 K. The adhesives include epoxy-nylon, nitrile-phenolic, aluminum filled epoxy-phenolic, epoxy-polyamide and polyurethane. The sealants include silicone, polyurethane and butadiene. Adherends were 301 and 321 stainless steel, 2024 aluminum alloy, titanium-5Al-2.5Sn alloy and some sandwich structure composites. The results show the superiority of the epoxy-nylon systems in all cases with nitrile-phenolic next. Of the sealants, the silicone compound had the highest shear strength. The author points out the rather low values of impact strength for all of the adhesives because of their inherent brittleness at cryogenic temperatures. In areas where their use allows, fillers may be developed to alleviate this problem. The author points to several problems yet to be thoroughly tested such as radiation effects, vacuum effects and thermal cycling.

Important references:

1. Hertz, J., Adhesives Age (Aug 1961).
2. Hertz, J., Advances in Cryogenic Engineering 7, Plenum Press, Inc., NY (1962).
3. Eppinger, C. E. and Love, W. J., Denver Research Institute, University of Denver, Advances in Cryogenic Engineering 4, Plenum Press, Inc. NY (1960).
4. McClintock, R. M. and Hiza, M. J., Modern Plastics, June 1958.

CRYOGENIC ADHESIVE EVALUATION STUDY

Hertz, J.

General Dynamics/Convair, San Diego, Calif., Rept. No. ERR-AN-032 (Jan 1961) 79 pp

Investigations were carried out to test the suitability of a group of epoxy-nylon adhesives for use down to 20 K. The evaluation was performed specifically for aerospace applications at liquid oxygen and liquid hydrogen temperatures. The adhesives were in the form of commercially available unsupported tape from three suppliers. No attempt was made to select the best of the supplied adhesives for cryogenic applications. The investigation included testing metal-to-metal adhesive-bonded joints with lap-shear, butt-tensile and impact specimens, plastic-to-plastic bonded joints with lap-shear coupons, and composite sandwich structures with pi-tension and plate-shear specimens. The lap-shear specimens were either EHF 301 CRES with Hexcel honeycomb core, Ti-5Al-2.5Sn alloy, or 2024 aluminum alloy. The butt-tensile and impact specimens were all stainless 321. Test temperatures were 298 K, 200 K, 77 K and 20 K. Comparisons were made with various other adhesives including epoxy, epoxy-phenolic, nitrile-phenolic, epoxy-polyamide and polyurethane. Tests were also conducted on the effects of surface preparation and thermal cycling; in the latter case the variability in individual samples was in the same range as the variability due to cycling. The data obtained in this study indicate that the epoxy-nylon class of adhesives is superior to all other classes commercially available at the time of the tests in the temperature range 20 K to 298 K. No attempt was made in this program to obtain design allowables and the type of joint and type of application are strong factors in the type of test needed. A less-detailed report of this study is given in a paper presented by Hertz at the 1961 Cryogenic Engineering Conference (see citation in Secondary Documents).

Important references:

1. Hertz, J., Adhesives Age (August 1961).
2. McClintock, R. M. and Hiza, M. J., Modern Plastics (June 1958).

AN EVALUATION OF THE MECHANICAL PROPERTIES OF ADHESIVES AT CRYOGENIC TEMPERATURES AND THEIR CORRELATION WITH MOLECULAR STRUCTURE

Hertz, J.

General Dynamics/Astronautics, San Diego, Calif., Rept. No. GDA-ERR-AN-196 (Sep 1962) 54 pp

New experimental data from the author are given in this report along with data summarized from previous cryogenic adhesive studies. The new data are tensile-shear, butt-tensile and impact tests. The shear tests used stainless steel 301, titanium-5Al-2.5Sn and an epoxy-fiberglass laminate as adherends. The butt-tensile and impact tests used stainless steel 321. The adhesives tested included epoxy-nylon, epoxy-polyamide, and epoxy. Most of the adhesives tests are reported using the commercial rather than the generic names of the adhesives. Test temperatures for the mechanical properties were 20 K, 77 K, 200 K and 298 K. The results are presented as functions of temperature, surface preparation and adherend material. The results show the nylon-epoxy systems having the highest strength at cryogenic temperatures as well as higher temperatures (two to three times the strength of epoxy-polyamide systems). This was true for all three mechanical property tests. The results of these tests correlated reasonably well with those of other studies. The report also includes results of thermal expansion measurements for the adhesives from 77 K to 400 K. Infrared spectrum curves are reported for each of the adhesives.

The report also contains results of pi-tension and shear tests of sandwich panels utilizing epoxy-nylon adhesives bonding either titanium alloy or stainless steel faces to a honeycomb core. These tests were conducted at 20 K, 77 K and 298 K. The results show that these composites have greater strength at lower temperatures and that the honeycomb core will fail before the adhesive.

Important references:

1. McClintock, R. M. and Hiza, M. J., CEL, NBS, WADC Technical Report 59-260 (Nov 1959).
2. Hiza, M. J. and Barrick, P. L., CEL, NBS, SPE Transactions (Apr 1961).
3. Hertz, J., Advances in Cryogenic Engineering 7, 336 (1961).
4. Smith, M. B. and Susman, S. E., Narmco Research and Development, Telecomputing Corp., Annual Summary Report, NASA Contract NAS 8-1565 (May 1962).

POLYURETHANE ADHESIVE AT -423°F

Hertz, J.

General Dynamics/Astronautics, San Diego, Calif., Rept. No. GDA-MRG-95
(Aug 1959) 2 pp

This brief report gives fatigue test results on a polyurethane adhesive at 20 K. Previous testing showed that the adhesive could be used to bond polyurethane foam to stainless steel, polyurethane foam to itself, or phenolic-fiberglass laminate to itself. Such bonds withstood cooling to 77 K and to 20 K with no apparent embrittlement. For a better evaluation of the adhesive, it was used to bond polyurethane foam to stainless steel, and the specimen was subjected to fatigue cycling while immersed in liquid hydrogen. Inspection after 1430 cycles at 20 K showed a crack in the foam but no apparent debonding. The foam had cracked again after 1700 cycles at 20 K. When the foam was removed, small areas were found adjacent to the cracks where the bond had broken. In all other areas, the bond was stronger than the foam. The polyurethane adhesive was recommended for bonding polyurethane foam to stainless steel for application at temperatures below 172 K.

LIQUID HYDROGEN TARGETS OF ADHESIVE-BONDED MYLAR PLASTIC

Hickman, R. S., Kenney, R. W., Mathewson, R. C., and Perkins, R. A.

(California Univ., Berkeley. Lawrence Radiation Lab.)

Rev. Sci. Instrum. 30, No. 11, 983-5 (Nov 1959)

A series of vacuum-tight liquid-hydrogen containers was fabricated for use as targets for particle accelerators. The structures were made of mylar sheet, sometimes shaped by hot molding, adhesively bonded to itself or to metals. Two adhesives, both epoxies, were found suitable for the bonding. Of the configurations illustrated, the bonds included mylar-mylar, mylar-brass, mylar-stainless steel, and mylar-copper. Burst pressures of these configurations were given, although specific bond strengths were not. Epoxy-bonded mylar-metal joints were satisfactory with brass, copper, or cadmium as the metal, less satisfactory with steel, borderline because of tendency to crack with stainless steel, and least satisfactory with aluminum because of adhesion difficulties. One adhesive was preferred for aluminum bonds. Much general information is included in the paper on forming the mylar, making bonds, and leak-testing structures. Another paper by Mehr and McLaughlin (see citation in Secondary Documents) gave even more-specific instructions on methods of building laminated-dome liquid hydrogen targets by these methods.

STATUS OF ADHESIVES RESEARCH AT MARSHALL SPACE FLIGHT CENTER

Hill, W. (National Aeronautics and Space Administration, Marshall Space Flight Center, Huntsville, Ala.)

Structural Adhesive Bonding Conference, National Aeronautics and Space Administration, Marshall Space Flight Center, Huntsville, Ala. (Mar 15-16, 1966) Paper 3, Session II, 15 pp

This paper briefly reviews the results of three research programs on cryogenic adhesives, carried out under NASA contracts. The first of these programs attempted to produce an adhesive system useful over a wide temperature range. Resins of several classes, poly-2-oxazolidones, polyisocyanurates, polyurethanes, bisphenol epoxies, and epoxy esters, were tested for tensile-shear strength at temperatures from 20 to 477 K. Of the classes tested, the polyoxazolidones and polyisocyanurates appeared to be most deserving of further investigation. The second program studied the effects of primers and of aging at room temperature and above on polyurethane adhesives. Aging at 310 K and high humidity produced rapid deterioration of tensile-shear strengths at 355 K and RT and somewhat less deterioration of strength at 89 K. Aging at 294 K caused less-severe deterioration. In all cases, primed specimens were superior to unprimed specimens, and a silane primer was superior to a polyester primer. The third program, for synthesis of polymers for adhesive systems insensitive to impact under liquid oxygen, was not discussed because of security classification. Details on the three programs reviewed are available in other sources. This review does not refer directly to sources of detailed results, but contract numbers are given.

COMPOSITE STRUCTURAL MATERIALS INVESTIGATION AT CRYOGENIC TEMPERATURES
Hillig, W. B., LeGrand, D. G., Mehan, R. L., deTorres, P. D., and Coblentz,
W. S.

Materials Research for Superconducting Machinery - III. National Bureau
of Standards, Boulder, Colo., Cryogenics Div. (Contract Monitor).
General Electric Co., Schenectady, N.Y. Semi-Annual Tech. Rept., Sep
1974 - Mar 1975, Rept. No. SRD-75-038. Sponsored by Advanced Research
Projects Agency, Arlington, Va., Order No. ARPA-2569 (Apr 1975) 41 pp

The purpose of the program is to develop systems for bonding fiber reinforced composites to structural metals for superconducting machinery. A literature survey was made on adhesive bonding for use at cryogenic temperatures. This excellent survey covers some 33 references, under the categories of solid-solid bonds, bonds involving thin layers, physics and chemistry of cryogenic adhesives, and reviews. The data in each reference is summarized in an extensive tabulation showing the objectives of the work, the adhesives, adherends, and temperature ranges involved, and some comments on results and conclusions. A brief discussion of the survey notes that epoxies, epoxy-nylons, and polyurethanes appear to be the best cryogenic adhesives, that they have their advantages and limitations, and that adhesive selection depends on application and environment since there is no single superior cryogenic adhesive. The results are said to be encouraging for the bonding of composites to metals, although available information is only qualitative.

The remainder of the report gives some preliminary results on bonding glass-fiber reinforced epoxy composite to 6061 aluminum at 4 K. Bonds are made by curing the composite between aluminum layers. A few shear tests at 4 and 298 K, to determine optimum aluminum surface treatment, are reported. The great value of this progress report is in the comprehensive literature survey, rather than the preliminary and incomplete experimental data.

Important references:

1. Smith, M. B. and Susman, S. E., Advances in Cryogenic Engineering 8, Edited by K. D. Timmerhaus, Plenum Press, N.Y. (1963).
2. Kausen, R. C., Mater. Design Eng. 60, 108 (1964).
3. Landrock, A. H., Plastic Tech. Eval. Center, Rept. 20 (1965) pp 253.
4. Codlin, E. M., Cryogenics and Refrigeration, 1F11 Plenum, N.Y. (1968).

CRYOGENIC ADHESIVE PROPERTIES OF BISPHENOL-A EPOXY RESINS

Hiza, M. J., and Barrick, P. L. (National Bureau of Standards, Boulder, Colo. Cryogenic Engineering Lab.)

SPE Trans. 1, No. 2, 1-7 (Apr 1961)

A series of studies was conducted to determine the performance characteristics of epoxy resin adhesive systems at low temperatures, and to provide some basis for formulation of a system with optimum cryogenic properties. The adhesives were bisphenol-A epoxides with molecular weights from 384 to 1808, used to make aluminum-aluminum lap joints and block joints, with meta-phenylenediamine used as curing agent. Experiments consisted of chemical determination of epoxide equivalents (for calculation of molecular weight); total thermal contraction of the cured resin in bulk between 300 and 76 K; and tensile-shear strengths of lap joints and impact-shear strengths of block joints at 76 and 300 K. At 300 K, tensile-shear strengths increased while impact-shear strength decreased with increasing molecular weight of resin. At 76 K, the tensile-shear strengths were maximum near a molecular weight of 600, with the average values for molecular weights of 384 and 634 exceeding the values at room temperature. Impact-shear strengths at 76 K were much lower than at 300 K and showed no definite trend with molecular weight. The total thermal contraction of the resin increased with increasing molecular weight. Preliminary tests using diethylaminopropylamine as curing agent showed strengths 30% lower at 300 K and 60% lower at 76 K than samples cured with meta-phenylenediamine. The authors conclude that the total thermal contraction of a resin between 300 and 76 K provides a useful index of residual stresses, and thus of tensile-shear strength, at temperatures below 76 K. Of the materials tested, the bisphenol-A epoxide with molecular weight of 634, using an aromatic amine cure, would produce the best adhesive, based on a compromise of tensile-shear and impact-shear strengths at all temperatures and of ease of handling.

Important references:

1. Epstein, G., Adhesive Bonding of Metals, p. 103-104, Reinhold Publ. Corp., New York, N.Y. (1954).
2. Dietz, A. G. H., Adhesion and Adhesive Fundamentals and Practice (a symposium) p. 218, John Wiley and Sons, Inc., New York, N.Y. (1954).
3. McClintock, R. M. and Hiza, M. J., Modern Plastics 35, No. 10, 172 (1958).
4. Frost, W. M., Paper presented at the Cry. Eng. Conf. at the Univ. of California, Berkeley, Calif. (1959).

ADHESIVE-LINED, SCREW-IN TYPE JOINTING OF ALUMINUM AND AUSTENITIC STEEL
FOR CONTROL VALVES IN LOW-TEMPERATURE PLANTS

Hofmann, A., and Michely, R.

Linde Rep. Sci. Technol. 21, 3-6 (1975)

A threaded joint sealed with adhesive was developed for use on valves in air-separation plants. The objective of the development was to reduce potential leakage by eliminating a flanged connection between an aluminum valve and a chrome-nickel-steel extension tube. The joint was designed so that the threads carry the load while adhesive on the threads prevents turning and acts as a sealant. An additional metal-metal sealing edge prevents contact of the working medium with the adhesive. An epoxy was selected as the adhesive, and applied to the threads of both metals in a model simulating a valve. After assembly and curing, the joint was leak tested with helium at pressures up to 8.3 MPa, first at 293 K, then at 77 K, followed by ten rapid temperature cycles between 77 and 403 K, and helium leak tests as before at 293, 77, and 373 K. The leakage of the adhesive-lined joint remained low, at the limit of measurement, except for some increase at 373 K and pressures between 5 and 8.3 MPa. A photomicrograph of a section through the joint showed uniform distribution of the adhesive in the threads, with numerous small pores or bubbles within the adhesive. The authors conclude that the joining method is applicable between two dissimilar metals at temperatures from 73 to 403 K.

LINERS FOR NON-METALLIC TANKS

Hoggatt, J. T., and Workman, L. J.

Boeing Co., Seattle, Wash., Aerospace Group, Final Rept., National Aeronautics and Space Administration Rept. No. NASA-CR-54868, Contract No. NAS3-7944 (Jan 1966) 54 pp

The purpose of this program was to evaluate a polyimide film material for use as a liner inside filament-wound non-metallic liquid hydrogen tanks. As part of the evaluation program, four adhesives and three joint concepts were evaluated for joining the polyimide film to itself. The adhesives were an epoxy, a polyurethane, and two polyesters, and the joint concepts were tapered lap joint, butt joint with doubler, and butt joint with reinforced doubler. Specimens of each joint concept, using each adhesive, and with two different polyimide film thicknesses, were tested for uniaxial tensile strength at 20 K. In these uniaxial tensile tests, all of the failures occurred in the bond area, but all were failures of the film rather than of the bond. The strength of the film in the bond area was only about 45% of the strength of the plain film. One of the polyester adhesives consistently produced higher joint strengths in all combinations, and was chosen for further evaluation. The polyurethane adhesive was selected as the alternate. The lap joint and the butt joint with reinforced doubler were selected for further tests. Diaphragms were fabricated with the two adhesives and the two joint concepts, and tested for biaxial tensile strengths (diaphragm burst tests) at 20 K. Test results led to selection of the polyester adhesive and lap joint as the best combination, and cylinders of the film were fabricated and enclosed in filament-wound epoxy cylinders as simulated tanks. The cylinders performed well in burst tests, but not in cyclic pressurization tests at 20 K. One of the cylinders failed in the bond area, but not in the bond itself. None of the liner failures could be attributed to failure of the adhesive joint. The polyimide film was deemed adequate as a liner for non-metallic liquid hydrogen tanks only if there is no requirement for cyclic loading. The loss of strength of the film in the bond area indicated the need for an improved bonding process.

Important references:

1. Hoggatt, J. T., Boeing Document D2-23778-1 (Jan 1965).
2. Toth, J. M., Sherman, W. C. and Soltysiak, D. J., Douglas Aircraft Company, NASA CR-54393 (Sep 1965).
3. Hanson, M. P., Richards, H. T. and Hickel, R. O., NASA TN D-2741 (1965).

OPTIMIZATION OF THE PERFORMANCE OF A POLYURETHANE ADHESIVE SYSTEM OVER THE TEMPERATURE RANGE OF -423°F TO $+200^{\circ}\text{F}$

Holland, H., McLeod, A. H., and Steele, D. (Whittaker Corp., San Diego, California)

Whittaker Corp., San Diego, Calif., Narmco Research and Development Div., Annual Summary Rept., National Aeronautics and Space Administration Rept. No. NASA-CR-84518, Contract No. NAS8-11958 (Mar 1967) 158 pp

In previous programs, a polyether-based polyurethane polymer was the most promising material for a cryogenic adhesive. Use of the adhesive in structural applications resulted in wide variations in bond performance, particularly peel strength. A research program was conducted to find the reasons for the variations in adhesive performance, and to develop methods of eliminating or minimizing the variations. Earlier work in the program showed that bond thickness affected peel strength at room temperature and above, that high humidity was the major factor in gradual degradation of bond strengths, and that a siloxane primer improved strength and decreased the effect of humidity. This report covers continuing research to improve the performance of the polyurethane adhesive. Tensile-shear and Bell peel tests of aluminum-aluminum bonds at 77 K, RT and 366 K were used to evaluate bond strengths. Various primers and application methods were tested, and siloxane-primed bonds showed improved strength, especially in combination with integral blending of some siloxane with the polyurethane adhesive. Humidity during storage caused severe initial loss of strength, then a levelling off over long-term storage, with siloxane-primed bonds remaining superior. Non-primed bond strength could be partially recovered by drying after storage at high humidity. Variations in adhesive formulation and bonding processes produced small or insignificant strength improvements. Variation of chemical structure of the polyurethane improved strengths at room and elevated temperatures. Pot lives could be controlled by changes in the curing agent. Greatest improvement as a cryogenic adhesive resulted with siloxane priming, siloxane blended with the adhesive, and elimination of the degassing in the bonding procedure.

ELECTRICAL LEADS INTO A HIGH-PRESSURE CHAMBER

Itskevich, E. S., Kraidenov, V. F., Mironova, Z. A., Slavyaninova, E. L., and Sukhoparov, V. A. (Academy of Sciences of the USSR, Moscow. Inst. of High-Pressure Physics)

Instrum. Exp. Tech. No. 1, 194-5 (Jan-Feb 1968), Transl. of Prib. Tekh. Eksp., No. 1, 187-8 (Jan-Feb 1968)

This note describes some Russian experience in using sealing compounds around electrical leads passing into a high pressure chamber. The sealing compounds were based on epoxy resins, for which only the Russian designations are given. The compounds were used to seal openings of 0.6 to 1 mm diameter, with 6 to 12 copper wires of 0.15 to 0.21 mm diameter passing through. The high pressure chambers with sealed openings were used repeatedly, at pressures up to 3 GPa at room temperature and 1.8 GPa at 77 and 4 K, without any cases of leakage through the seals. Some variations of the sealing compound were tested, including one with 22 weight percent boron nitride powder filler and another with 20 weight percent alumina filler. The variations also performed without failure, although tests were run only up to 1.5 GPa.

Important references:

1. Balla, D. and Brandt, N. B., Zh. Eksperim. i Teor. Fiz. 47, 1653 (1964).

SUMMARY OF MATERIALS TECHNOLOGY OF M-1 ENGINE

Janser, G. R. (Aerojet-General Corporation, Sacramento, California)
Aerojet-General Corp., Sacramento, Calif. Liquid Rocket Operations,
Rept. No. AGC-8800-64, National Aeronautics and Space Administration
Rept. No. NASA-CR-54961, Contract No. NAS3-2555 (Jul 1966) 85 pp

This report is a compilation of a number of engineering tasks of limited scope performed in support of the M-1 Engine Program. One such task was to evaluate materials to fill the space between the engine thrust chamber and its reinforcing jacket. Among the requirements for the material were flexibility during cool-down, high yield strength at cryogenic temperatures, and thermal expansion values near those of the jacket material.

Four filled-epoxy resin adhesive systems, ranging from flexible to rigid, were evaluated. The three more flexible systems were aluminum-filled, while the rigid system was filled with aluminum and asbestos. Lap-shear strengths of aluminum-aluminum bonds with the four adhesives were determined at 77 and 293 K. Cured bulk samples of the adhesives were tested for compressive strength and percent compressive strength and percent compressive strain at failure at 77 and 293 K. The most flexible adhesive was superior except in room-temperature compressive strength. This adhesive was compared with the rigid epoxy in further tests, for tensile strength and elongation of cured bulk samples at 77 and 293 K, and for coefficient of thermal expansion of machined and of molded specimens between 77 and 293 K. The rigid adhesive had a higher tensile strength at room temperature, while the flexible adhesive was better at 77 K. The greater flexibility of the flexible material was shown by the elongation at both temperatures. The rigid adhesive had the lower thermal expansion coefficient. The flexible adhesive was again tested for lap-shear strength, this time with Inconel 718 adherends, at 77 and 298 K, with variations in cure cycle. A shorter, higher-temperature cure gave the better bond strengths. The adhesive was also tested for its flow characteristics during cure, by determining distance of flow on steel panels held at 60 degrees or 90 degrees from horizontal, for varying adhesive thicknesses.

THE EFFECT OF THERMAL SHOCK ON ADHESIVE BONDS

Jeffs, A. T. (Rocket Propulsion Establishment, Westcott, England)
Spaceflight 8, No. 9, 335 (Sep 1966)

This paper presents results of some tests of adhesive bonds between thin metal plates. The tests were conducted to determine the resistance to thermal shock of various adhesives. The thermal cycling (shock) was done by successive immersion in boiling water followed by liquid hydrogen (373-20 K), liquid nitrogen (373-77 K) and solid carbon dioxide cooled acetone (373-195 K). The metals used were an aluminum alloy, a stainless steel (EN48B) and two titanium alloys (317 and 318A). Four adhesives were tested: A) aluminum powder filled epoxy, B) nylon filled epoxy, C) non-filled epoxy and D) an unidentified adhesive. In each thermal cycle test the authors report an average room temperature tensile-shear strength value after the thermal cycling. Adhesives C) and D) exhibited rather low strengths and gave either degraded results or failed after thermal cycling. Adhesives A) and B) showed little change in strength after thermal cycling and in some cases even gave high strengths after cycling.

On the basis of these results the author concludes that adhesively bonded joints should withstand severe thermal shock and have excellent bond integrity with several high strength alloys. The filled adhesives are more suitable for cryogenic service with or without thermal shock.

THE SPECIFIC HEAT OF THE GLUE BF-2 BETWEEN 0.3 AND 4.2 DEGREES K

Kalinkina, I. N.

Instrum. Exp. Tech. No. 1, 163 (Jan-Feb 1958). Transl. of Prib. Tekh. Eksp., No. 1, 146 (1958)

This note gives the results on specific heat measurements of a Russian adhesive. The adhesive is often used for structural bonds or electrical insulation, in low-temperature experiments where its contribution to total specific heat must be known to obtain accurate results. The adhesive, identified only by its Russian designation, was applied in several layers to a thin copper foil, specific heats were measured over the range from 0.3 to 4.2 K, and the specific heat of the copper foil was subtracted from the total. Results are presented graphically, showing a linear relationship between specific heat and T^3 . A simple equation describes the specific heat of the adhesive from 0.3 to 4.2 K within 3 to 5%.

ADHESIVES FOR HIGH AND LOW TEMPERATURES

Kausen, R. C. (Little (Arthur D.), Inc., Cambridge, Mass.)

Mater. Des. Eng. 60, No. 3, 108-12 (Sep 1964)

This is a two-part review of the advantages and limitations of the different types of structural adhesives for use in the temperature range 20 K to 1100 K. The first part covers high temperature, and the second part cryogenic temperatures as well as a summary of promising new formulations. The systems reviewed are polyurethanes, epoxy-nylon, epoxy-phenolics, rubber-phenolics, vinyl acetal-phenolics, epoxy-polyamides, filled epoxies and polyimides. The author includes a very useful table which lists for each system the following information: performance (temperature) range, advantages, limitations, form available, applications and name, number and source of commercially available varieties. The paper contains graphs comparing the various systems on the basis of shear-tensile strength versus temperature as well as qualitative information on such things as peel strength and impact testing (brittleness). The paper points out the overall best performance of polyurethanes as long as upper temperatures are kept below about 360 K. Epoxy-nylons have the same upper temperature limitation (in fact an even lower temperature range), however they are widely used due to light weight and easy availability. Epoxy-phenolics have a wide temperature range, but have a fairly low peel strength. Epoxy-polyamides are used frequently even though their low temperature strength is about half that of polyurethanes and epoxy-nylons. Their ease of use makes them attractive where high strength is not needed. Filled epoxies (unmodified with no secondary resin) offer advantages where thermal expansion needs to be controlled. The filler can be anything from glass fibers to metal powder. The author discusses the potential of polyimides since they retain flexibility even down to 20 K.

Important references:

1. Frost, W. M., Advances in Cryogenic Engineering 5 (Proceedings of Cryogenic Engineering Conference, University of Calif., Sep 1959).
2. Hertz, J., Adhesives Age (Aug 1961).
3. Hertz, J., Advances in Cryogenic Engineering 7 (Plenum Press Proceedings of Cryogenic Engineering Conference, University of Michigan, Aug 1961).
4. Kuno, J., SAMPE (May 1964).

HIGH- AND LOW-TEMPERATURE ADHESIVES--WHERE DO WE STAND?

Kausen, R. C. (Little (Arthur D.), Inc., Cambridge, Mass.)

Materials Symposium (Proc., National SAMPE Symposium on Adhesives and Elastomers for Environmental Extremes, 7th, Los Angeles, Calif., May 20-22, 1964), Sect. 1, 1-51, Society of Aerospace Material and Process Engineers (1964)

The state-of-the-art in high and low temperature structural adhesives, circa 1964, is summarized in this paper. The author makes the valid point that he is evaluating the published results from the standpoint of a user of adhesives rather than from the point of view of a developer of proprietary products. The paper includes a list of manufacturers as well as a generic-commercial designation -supplier table for all of the adhesives surveyed. The extensive bibliography contains 221 references. The paper is divided into two parts: high temperature adhesives and low temperature adhesives; this abstract covers only the latter. The low temperature (cryogenic) adhesives covered include epoxy, epoxy-phenolic, epoxy-nylon, epoxy-polyamide, vinyl acetal-phenolic, rubber-phenolic and polyurethane. Generalized strengths from 20 K to 400 K are given for each generic type. From the standpoint of shear strength at cryogenic temperatures epoxy-nylon and polyurethane offer the highest strength, but the designer should select carefully depending on the temperature range. Low and high temperature strengths are certainly not the same and the strength of polyurethane, for example, falls off rapidly above room temperature.

Important references:

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2. Hertz, J., Advances in Cryogenic Engineering 7, Plenum Press Proceedings of 1961 Cryogenic Engineering Conference, University of Michigan, Ann Arbor, Aug 15-17, 1961, Edited by K. D. Timmerhaus (1962).
3. Hertz, J., Adhesives Age 4 (8), 3037 (Aug 1961).

ADHESIVE BONDING OF NICKEL AND NICKEL-BASE ALLOYS

Keith, R. E., Monroe, R. E., and Martin, D. C. (Battelle Memorial Inst., Columbus, Ohio)
National Aeronautics and Space Administration, Marshall Space Flight Center, Huntsville, Ala. Tech. Memo. NASA-TM-X-53428. Army Missile Command, Redstone Arsenal, Ala., Redstone Scientific Information Center Rept. No. RSIC-480, Contract No. DA-01-021-AMC-11651(Z) (Oct 1965) 69 pp

This report is a state-of-the-art review of adhesive bonding of nickel and nickel alloys. After some general comments on adhesive bonding, the review covers such subjects as joint design, adherend surface preparation, selection of the adhesive, application of the adhesive, joint assembly, curing, cleaning, testing, and inspection. Consideration of inorganic adhesives and an extensive bibliography of 72 references complete the review. Part of the section on selection of the adhesive deals with cryogenic temperature service conditions, by reviewing data from eight references. The authors stress that this data is useful only as a rough guide, because the adherends were not nickel or nickel-based alloys. Relatively little work has been done on adhesive bonding of the nickel-based alloys because they are often used at temperatures well above maximum service temperatures of organic adhesives or under corrosive conditions. For cryogenic service, polyurethanes will probably be satisfactory, but the need for further research is pointed out.

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3. Hertz, J., Convair-Astronautics, San Diego, California, Report ERR-AN-032, Third Quarterly Progress Report, Phase I, Contract AF 33(616)-7984, AD 273219 (Jan 25, 1961) (RSIC 0020).
4. Frost, W. M., National Bureau of Standards, Boulder, CO, Rept. WADC-TR-59-260, Contract DO 33(616)-58-12 (Nov 1959) (RSIC 0015).
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6. Miller, R. N., Bailey, C. D., Freeman, S. M., Beall, R. T., and Coxe, E. F., Industrial and Engineering Chemistry 1 (4) 257-61 (Dec 1962) (RSIC 0022).
7. Smith, M. B., and Susman, S. E., Whittaker Corp., Narmco Research and Development Division, San Diego, Calif., Summary Rept., Contract NAS-8-1565 (Jul 15, 1962) (RSIC 0276).
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ADHESIVE BONDING OF TITANIUM AND ITS ALLOYS

Keith, R. E., Monroe, R. E., and Martin, D. C. (Battelle Memorial Inst., Columbus, Ohio)

National Aeronautics and Space Administration, Marshall Space Flight Center, Huntsville, Ala., Tech. Memo. NASA-TM-X-53313. Army Missile Command, Redstone Arsenal, Ala., Redstone Scientific Information Center Rept. No. RSIC-414, Contract No. DA-01-021-AMC-11651(Z) (Aug 1965) 96 pp

This report is a state-of-the-art review of adhesive bonding of titanium and titanium alloys. After some general comments on adhesive bonding, the review covers such subjects as joint design, adherend surface preparation, selection of the adhesive, application of the adhesive, joint assembly, curing, cleaning, testing, and inspection. Some applications of adhesive-bonded titanium alloys and an extensive bibliography of 91 items complete the review. Part of the section on selection of the adhesive deals with cryogenic temperature service conditions, by reviewing data from eight references. The authors stress that most of this data is useful only as a rough guide, because the adherends were not titanium or titanium alloys. The data which are of direct application came from Hertz (tensile-shear strengths of epoxy-nylon, nitrile-phenolic, epoxy-phenolic, and epoxy-polyamide adhesives, Ti-8Mn adherends, 20 to 300 K), from Arslett and Jeffs (room temperature tensile-shear strengths with epoxy adhesives, Ti-5Al-2.5Sn and Ti-6Al-4V adherends, after cycling from room temperature to 77 or 20 K), and from Smith and Susman (tensile-shear strengths of epoxy-polyamine with nylon, epoxy-polyamine with FEP films, and polyurethane adhesives, Ti-8Mn adherends, 77 K and room temperature).

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ADHESIVE BONDING OF STAINLESS STEELS--INCLUDING PRECIPITATION-HARDENING
STAINLESS STEELS

Keith, R. E., Randall, M. D., and Martin, D. C. (Battelle Memorial
Inst., Columbus, Ohio)

National Aeronautics and Space Administration. Marshall Space Flight
Center, Huntsville, Ala., Spec. Publ. NAS-SP-5085. Army Missile Command,
Redstone Arsenal, Ala., Redstone Scientific Information Center Rept. No.
RSIC-599, Contract No. DA-01-21-AMC-11651(Z) (1966) 125 pp

This report is a state-of-the-art review of adhesive bonding of stainless steels. After some general comments on stainless steels and on adhesive bonding, the review covers such subjects as joint design, adherend surface preparations, selection of the adhesive, application of the adhesive, joint assembly, curing, cleaning, testing, and inspection. Consideration of inorganic adhesives, some applications of adhesive-bonded stainless steel, and an extensive bibliography of 110 items complete the review. Part of the section on selection of the adhesive deals with cryogenic temperature service conditions, by reviewing data from eleven references. The authors stress that much of this data is useful only as a rough guide, because the adherends were not stainless steel. The data which are of direct application came from Litvak, Aponyi, and Tomashot (tensile-shear strength of PBI adhesive, 17-7 PH stainless steel adherends, 20 to 810 K), from Reinhart and Hidde (fatigue strength of PBI adhesive, PH 15-7 Mo adherends, 77 to 645 K), and from Arsllett and Jeffs (room-temperature tensile-shear strengths with epoxy adhesives, stainless steel and titanium alloy adherends, after cycling from room temperature to 77 or 20 K).

Important references:

1. Kausen, R. C., Adhesives for High and Low Temperatures, Paper presented at the 7th National SAMPE Symposium, Los Angeles, Calif. (May 20-22, 1964) (RSIC 0375).
2. Kuno, J. K., Comparison of Adhesive Classes for Structural Bonding at Ultra-high and Cryogenic Temperature Extremes, Paper presented at the 7th National SAMPE Symposium, Los Angeles, Calif. (May 20-22, 1964) (RSIC 0281).
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4. Reinhart, T. J., Jr. and Hidde, R., Mechanical Properties of Imidite Adhesives, in Symposium on Structural Adhesives Bonding, at Stevens Institute of Technology, sponsored by Picatinny Arsenal and Stevens Institute of Technology, Sep 14-16, 1965.
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6. Frost, W. M., National Bureau of Standards, Boulder, Colorado, Report WADC-TR-59-260, Contract DO 33(616)-58-12 (Nov 1959) (RSIC 0015).
7. Smith, M. B. and Susman, S. E., SAE Journal 71, 38-41 (May 1963) (RSIC 0382).
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9. Smith, M. B. and Susman, S. E., Whittaker Corporation, Narmco Research and Development Division, San Diego, Calif., Summary Report, Contract NAS-8-1565 (Jul 15, 1962) (RSIC 0276).
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MEASURED EFFECTS OF THE VARIOUS COMBINATIONS OF NUCLEAR RADIATION,
VACUUM, AND CRYOTEMPERATURES ON ENGINEERING MATERIALS. VOLUME II:
RADIATION-CRYOTEMPERATURE TESTS

Kerlin, E. E. and Smith, E. T.

General Dynamics/Fort Worth, Tex., Ann. Rept. No. FZK-188-2, National
Aeronautics and Space Administration Rept. No. NASA-CR-58830, Contract
No. NAS8-2450 (May 1964) 286 pp

This report gives detailed results from a series of tests to measure the combined effects of nuclear radiation and cryogenic temperatures on a group of nonmetallic materials proposed for use in the NERVA nuclear rocket program. The group of materials tested consisted of adhesives, sealants, seal materials, thermal and electrical insulation, structural composites (laminates), potting compounds and dielectrics. The measurements included tensile-shear strength, T-peel strength, leakage, ultimate tensile strength, ultimate elongation, stress-strain characteristics, thermal conductivity, and pull-out strength of potted wire. The test procedure was designed so that irradiation and property testing could be performed at liquid nitrogen temperature without warm-up. The properties were measured as a function of integrated neutron flux and gamma dose. Tests were also conducted with irradiation and testing at ambient conditions. Three radiation doses were used - zero, relatively low (10^9 ergs/gm) and relatively high (10^{10} ergs/gm).

The two adhesives tested were an epoxy-nylon and an epoxy-phenolic. The two sealant materials were an RTV silicone and a proprietary compound. The results of the tensile-shear tests for the epoxy-nylon adhesive showed a severe degradation after high dose irradiation at ambient temperature, and exposure to 77 K reduced the strength even further. It is not considered useful at 77 K in a radiation environment. The epoxy-phenolic showed very little radiation degradation or temperature reduction of tensile-shear strength although the initial strength was less than the epoxy-nylon.

With regard to the sealants, the proprietary compound fractured on cooling to 77 K and was judged unacceptable and not tested further. The RTV silicone sealant had a T-peel strength only 1/4 of the unirradiated samples after exposure to a high dose at ambient conditions. The 77 K test results were acceptable in the unirradiated conditions, but no values could be determined after irradiation. Most of these were adhesive failures so that further testing may prove these materials acceptable.

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1. Kerlin, E. E., and Smith, E. T., Annual Report (9 Nov 1962 thru 30 Apr 1964), GD/FW Report FZK-188-1 (1 may 1964).
2. Smith, E. T., Annual Report (9 Nov 1961 thru 8 Nov 1962), GD/FW Report FZK-161-2 (5 Jan 1963).
3. Kerlin, E. E., Annual Report (9 Nov 1961 thru 8 Nov 1962), Vol. I, GD/FW Report FZK-161-1 (5 Jan 1963).
4. Kerlin, E. E. and Smith, E. T., Quarterly Progress Report (1 Mar 1963 thru 31 May 1963), GD/FW Report FZK-167.

MEASURED EFFECTS OF THE VARIOUS COMBINATIONS OF NUCLEAR RADIATION,
VACUUM, AND CRYOTEMPERATURES ON ENGINEERING MATERIALS

Kerlin, E. E., and Smith, E. T.

General Dynamics/Fort Worth, Tex. Nuclear Aerospace Research Facility,
Rept. No. FZK-290, National Aeronautics and Space Administration Rept.
No. NASA-CR-77772, Contract No. NAS8-2450 (Jul 1966) 520 pp

This report summarizes the work done during the last two years of a five year contract with NASA to measure the effects (singly and in combination) of nuclear radiation, vacuum and cryogenic temperatures on structural adhesives, structural laminates, potting compounds, electrical and thermal insulation, dielectrics, thermal control coatings, seals, sealants and lubricants. The testing was done for the purpose of establishing guidelines in the selection of materials for the space Nuclear Propulsion System (NERVA). Tests were conducted in air at ambient temperatures, in liquid nitrogen at 77 K and in liquid hydrogen at 20 K. The tests included mechanical and tensile properties (measured at test temperature and in test environment), lubricating properties, electrical properties, and thermal conductivity of foam insulation.

The adhesives tested included nylon-epoxy, epoxy, polyurethane and epoxy-phenolic. The adhesives are all identified by commercial trade designations, but not by generic class. Most of the adhesives exhibited higher strengths at cryogenic temperatures than at ambient temperatures. Epoxy-nylon and polyurethane adhesives seemed least affected by nuclear radiation. Two sealants, identified only by commercial trade designation, were tested in air and vacuum with irradiation at ambient temperatures. Both of these materials exhibited marked degradation at all but the lowest radiation levels.

This report contains a large amount of useful data, but there is very little analysis of the results and no detailed conclusions are drawn generally or with regard to specific material classes.

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2. Smith, E. T., General Dynamics/Fort Worth Report FZK-161-2 (5 Jan 1963).
3. Kerlin, E. E. and Smith, E. T., General Dynamics/Fort Worth Report FZK-188-1 (1 May 1964).
4. Kerlin, E. E. and Smith, E. T., General Dynamics/Fort Worth Report FZK-188-2 (1 May 1964).

EFFECT OF LIQUID NITROGEN DILUTION ON LOX IMPACT SENSITIVITY

Key, C. F., and Gayle, J. B. (National Aeronautics and Space Administration, Marshall Space Flight Center, Huntsville, Ala.)

J. Spacecr. Rockets 3, No. 2, 274-6 (Feb 1966)

Organic materials such as polymeric adhesives in contact with liquid oxygen constitute fire and/or explosion hazards. The purpose of this study was to determine the effect of oxygen concentration on the impact sensitivity of various polymeric materials with liquid oxygen concentration varying from 100 percent to 20 percent (liquid air). The tests were conducted using the Army Ballistic Missile Agency (ABMA) Impact Tester with which a known weight (9.04 kg) is dropped from known heights (up to 1.1 m). The highest energy level that is withstood without reaction in 20 trials denotes the relative hazard associated with the material under test. The materials tested included laminates, honeycomb, silicone rubber, polyethylene terephthalate, polyurethane insulation, two foam insulations, a foam-filled honeycomb insulation, a rubber sealant, nylon-epoxy adhesive and HT-424 adhesive. The HT-424 exhibited acceptance (0% reaction frequency at 10 kg-m impact) at 20 percent oxygen, but not at higher concentrations. The nylon-epoxy adhesive was acceptable at concentrations of 50-60 percent oxygen and below. The rubber sealant was also found to be acceptable in the range 50-60 percent oxygen and below. Most materials, including the highly sensitive materials, were found non-reactive at 20 percent oxygen (liquid air). Several materials, however, exhibited reactions at slightly higher concentrations (30/70), including HT-424.

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2. Key, C. F. and Gayle, J. B., NASA TM X-53208 (Feb 15, 1965).
3. Lucas, W. R. and Riehl, W. A., Am. Soc. Testing Mater. Bull. 244, 29-34 (Feb 1960).

STRAIN GAGE APPLICATIONS ON EPOXY LAMINATES FOR USE AT CRYO TEMPERATURES
Kocher, R. M., and Johansson, J. W. (Lockheed Missiles and Space Co.,
Sunnyvale, Calif.)
ISA Annual Conference and Exhibit, 21st, New York (Oct 24-7, 1966) Paper
No. 16.14-1-66, 19 pp

Measurement of strain in thin laminates used as vapor barrier material in cryogenic insulation systems is often required to evaluate the design and to verify the design assumptions. Such measurements, however, are not altogether a routine test operation. This paper evaluates a number of commercially-available strain gages and adhesives required to bond them to the composites under test. Three types of laminates were used - polyethylene terephthalate film, polyester cloth and quartz cloth. Six adhesives and four types of strain gages were evaluated. The six adhesives consisted of one polyurethane and five epoxies. All of the six adhesives are identified by manufacturer and product number.

The specimens were strained to failure on both the polyethylene terephthalate film and the polyester cloth. Data were also taken on sensitivity versus microstrain. On the basis of the tests one type of epoxy was selected for the polyester cloth and another epoxy for the polyethylene terephthalate. The tests were performed while immersed in liquid hydrogen at 20 K.

MECHANICAL PROPERTIES OF FOAM MATERIALS IN THE TEMPERATURE RANGE OF 300°K TO 20°K (MECHANISCHE EIGENSCHAFTEN VON SCHAUM-STOFFEN IM TEMPERATURBEREICH VON 300°K BIS 20°K)

Kreft, H., and Wagner, D. (Bremen, ERNO Raumfahrttechnik G.m.b.H.)
Kaeltech.-Klim. 21, No. 9, 258-65 (Sep 1969)

Experimental results are given of an extensive investigation of the components, separately and together, of foam insulation systems for the upper stages of liquid hydrogen/liquid oxygen fueled rockets. The foam materials tested were polyurethane and polyvinylchloride, the protective film was aluminized mylar; and the adhesives were polyurethane, silicone, epoxy and a proprietary single component resin (results are shown only for the first two). All of the adhesives, as well as all of the other materials were identified as to manufacturer and product. Tests were conducted at 20 K, 77 K (or 80 K) and 293 K.

The foam insulation material was tested in tension and compression. Shear-tensile and T-peel tests were conducted on the adhesives, both with and without an intervening film of aluminized mylar. In addition, shear-tensile and T-peel tests were conducted where a foam sample was bonded between two layers of adhesive. The room temperature shear tensile strength of the silicone adhesive is less than one-third of the strength of the polyurethane adhesive, but the 77 K and 20 K strengths of the silicone are substantially higher than the polyurethane. The same is true with the film present except that the presence of the film (25 μ) reduces the strength 20 to 50 percent. In the tensile shear tests with the foam layer present, the breaking strength is essentially that of the foam since the adhesive strength is much greater. In the T-peel strength tests the silicone adhesive is again a little better at cryogenic temperatures compared with the polyurethane. The presence of the film and foam layers does not appreciably change the results although there is some reduction in both cases. With regard to the foams themselves the polyvinylchloride exhibits a tensile strength 4 to 5 times that of polyurethane foam but both show reductions at lower temperatures and the PVC advantage is reduced. The compressive strength of PVC is also higher than polyurethane foam.

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1. Kreft, H., Technischer Bericht RT/P-1/1/66, ERNO-Raumfahrttechnik GmbH, Bremen, Jan 1966.
2. Bäumler, F., Technischer Bericht 67/So/310, Linde AG, München, Aug 1967.
3. Bäumler, F., Technischer Bericht 68/So/68, Linde AG, München, Apr 1968.

SPECIFIC HEAT OF TEFLON-40 AND BF-2 GLUE IN THE 10-300 K TEMPERATURE RANGE

Krylovskii, V. S., Ovcharenko, V. I., and Khotkevich, V. I. (Kharkov State Univ., USSR)

J. Eng. Phys. (USSR) 22, No. 4, 450-1 (Apr 1972) (Transl. of Inzh. Fiz. Zh. 22, No. 4, 656-7 (Apr 1972))

Two polymeric materials which are often used in equipment for low-temperature experiments were measured. One of the materials was an adhesive, identified only by its Russian designation. It was applied in several layers to a copper foil, specific heats were measured from 20.3 to 284.5 K, and the specific heat of the copper was subtracted from the total. Results are presented in tabulated and graphical forms, and show a nearly linear increase of specific heat with temperature above 100 K. In earlier work by Kalinkina (see abstract on page 50), the specific heat of this same adhesive increased linearly with T^3 at temperatures below 4.2 K.

Important references:

1. Kalinkina, I. N., Pribory i Tekh. Eksperim., No. 1, 146 (1958).

COMPARISON OF ADHESIVE CLASSES FOR STRUCTURAL BONDING AT ULTRAHIGH AND CRYOGENIC TEMPERATURE EXTREMES

Kuno, J. K. (Whittaker Corp., Costa Mesa, Calif. Narmco Materials Div.)
Materials Symposium (Proc., National SAMPE Symposium on Adhesives and
Elastomers for Environmental Extremes, 7th, Los Angeles, Calif., May 20-
22, 1964) Sect. 11, 1-36, Society of Aerospace Material and Process
Engineers (1964)

This paper reviews the types and the capabilities at temperature extremes of a series of commercial adhesives. The adhesives are all products of a single company, Narmco Materials Div. of Whittaker Corporation. The materials claimed to be useful at cryogenic temperatures include a polyaromatic, an epoxy-phenolic, two modified epoxies, a phenolic-neoprene, three nylon-epoxies, an epoxy-polyamide, an epoxy-nylon-polyamide, and a polyurethane. Limited comparative data are given on tensile shear stresses of the adhesives at 20 K, and graphical comparisons cover various temperature ranges from 20 to 850 K. The discussion covers some of the general effects of the temperature extremes, some general advantages and disadvantages of adhesive bonding, and some of the design considerations and fabrication techniques. Usefulness of the review is somewhat limited, but it does provide some useful comparisons between classes of adhesives. More detailed information on these products is available in other papers.

CRYOGENIC PROPERTIES

Landrock, A. H. (Plastics Technical Evaluation Center, Dover, N.J.)
Encyclopedia of Polymer Science and Technology 4, 415-49, John Wiley and Sons, Inc., New York (1966)

This article on cryogenic properties of plastics and related materials includes a four-page review of cryogenic adhesives. The review, although brief, is very comprehensive, and represents the state of the art in about 1965. Cryogenic adhesives are defined and their major uses are listed. The use of RTV silicones as cryogenic sealants and non-structural adhesives is described. Stress concentrations and gradients within bonds are described as a major cause of problems associated with bonded joints, and the sources of such stress concentrations and means of minimizing their effects are discussed. The study of a number of adhesives, reported by Hertz (see abstract on page 37) in 1961, is reviewed, and a table of tensile-shear strengths of ten adhesives from this study is presented (four epoxy-nylons, one modified epoxy-phenolic, two nitrile-modified phenolics, a polyurethane, and two epoxy-polyamides, with aluminum, stainless steel, titanium, and reinforced plastic laminate adherends, at 20, 77, 200, and 299 K). Later work by other authors on polyurethanes, epoxy-nylons, the effects of fillers and film-supporting media and surface preparation, fluorinated ethylene-propylene copolymer films, and polybenzimidazole resin systems is reviewed less extensively, giving conclusions rather than numerical data. This review of cryogenic adhesives cites sixteen references.

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1. Kausen, R. C., Proc., 7th Natl. Symp., Adhesives and Elastomers for Environ. Extremes, Soc. Aerospace Mater. Process Engrs (SAMPE), Los Angeles, Calif., May 20-22, 1964, Sect. 1, pp 51.
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7. Smith, M. B. and Susman, S. E., Advances in Cryogenic Engineering 8, 300-305, Plenum Press, New York (1963).
8. Roseland, L. M., Proc., 7th Natl. SAMPE Symp., Adhesives and Elastomers for Environ. Extremes, Los Angeles, Calif., May 20-22, 1964, Sect. 7, pp 17.

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13-14, 1964, pp 87-103.
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Adhesives Bonding, Stevens Institute of Technology, Hoboken, NJ,
Sep 14-16, 1965, pp 534-584.

PROPERTIES OF PLASTICS AND RELATED MATERIALS AT CRYOGENIC TEMPERATURES

Landrock, A. H.

Plastics Technical Evaluation Center, Dover, N. J., Plastec Rept. No. 20
(Jul 1965) 253 pp

This report reviews the state-of-the-art of the effects of cryogenic temperatures on polymers. The report includes the following sections: molded plastics, thermal insulation, structural plastic laminates, elastomers, seals and sealants, adhesives, films and vapor barriers, fibers, electrical applications, friction and wear, liquid oxygen compatibility and radiation effects. The bibliography contains 302 annotated references, a detailed index to both the report and the bibliography, a conference index, a report number/corporate source index, a journal index and an author index.

Because of the paucity of available information on sealants at cryogenic temperatures, this review barely discusses the subject and gives references to five reports.

The report does nicely summarize the available (as of 1965) information on commercially-available adhesives down to 20 K. The summary section on adhesives covers twelve pages and includes tabular and graphical data, extracted from the literature on such generic types of adhesives as filled and unfilled epoxy, epoxy-nylon, epoxy-phenolic, nitrile-phenolic, epoxy-polyamide, rubber-phenolic, vinyl-phenolic, polyaromatic and polyurethane. The adhesives section does not draw any conclusions other than stating those found in the literature or comparisons drawn in the literature. The superiority of polyurethane for applications from about 400 K to 20 K was repeated from previous studies. Manufacturers' names and commercial designations for adhesives are included in this report. This report serves as a good summary of the state-of-the-art in 1965.

Important references:

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2. Hertz, J., ERR-AN-032, 25 January 1961. 74 pp. Contract AF33(616)-7984 (Compilation of Materials Research Data). (AD 273 219). CEL-NBS 11855.
3. Hertz, J., MRG-275, 21 November 1961. 25 pp. Contract AF 33(616)-7984 (Compilation of Materials Research Data). (AD 291 521). CEL-NBS 15855.
4. Hertz, J., *Adhesives Age* 4 (8): 30 (August 1961). CEL-NBS 16195.

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9. Lantz, R. B., Proceedings of the Sixth National SAMPE Symposium, Materials for Space Vehicle Use, held at Seattle, 19-20 November 1963. Vol. 2. Douglas Engineering Paper No. 1750.
10. McClintock, R. M. and Hiza, M. J., Modern Plastics 35 (10): 172 (June 1958); Advances in Cryogenic Engineering 3, 305-315 (1960). CEL-NBS 08907.
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14. Roseland, L. M., Proceedings, 7th National Symposium on Adhesives and Elastomers for Environmental Extremes, held by the Society of Aerospace Materials and Process Engineers (SAMPE) at Los Angeles, CA, May 20-22, 1964. Sect. 7, pp 17.
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MATERIALS FOR FILAMENT WOUND CRYOGENIC PRESSURE VESSELS

Lantz, R. B.

Douglas Aircraft Co., Inc., Santa Monica, Calif., Engineering Paper No. 1750. Presented in Materials for Space Vehicle Use (Proc. National SAMPE Symposium, 6th, Seattle, Wash., November 18-20, 1963) Vol 2, 29 pp, Society of Aerospace Material and Process Engineers (1963)

Adhesive systems were investigated as part of a program to develop filament wound pressurized cryogenic storage tanks. Adhesives bond the impermeable integral liner to itself and to the composite tank wall, and bond the end fitting to the vessel for a domed pressure vessel. Liners are either metallic or organic films, and the tank walls are a fiberglass composite material. Three adhesives were evaluated, a polyurethane and two modified epoxies. Tensile-shear strengths were found at five temperatures from 20 to 395 K, with unspecified adherends. The polyurethane had superior bond strengths from 20 to 200 K. The polyurethane and one modified epoxy were tested for tensile-shear strengths with organic film adherends at 20 K and RT. Three organic films were used, a polyester film, a polyvinyl fluoride film, and a polyimide film. With the polyester and polyvinyl fluoride adherends, the epoxy adhesive had the higher bond strengths at room temperature and the polyurethane was superior at 20 K. The epoxy adhesive had higher strengths at both temperatures with the polyimide film, and this combination produced the highest bond strengths observed in the tests. The authors conclude that the polyurethane adhesive shows the greatest promise as a cryogenic adhesive for application in filament wound pressure vessels, but the epoxy adhesive is included in a list of materials still being considered.

SUPERINSULATION RESEARCH AND DEVELOPMENT PROGRAM

Leonhard, K. E., Clay, J. P., Getty, R. C., and Tatro, R. E.

General Dynamics/Convair, San Diego, Calif., Rept. No. GDC-ERR-AN-1037
(Dec 1966) 197 pp

An adhesive system was required to make vapor tight seams in fabricating vapor barriers for an insulation system. Adhesives which performed satisfactorily in the laboratory generally lost their vapor seal properties while maintaining a mechanical bond when cooled to cryogenic temperatures. Since mylar had the high strength and low permeability to make a good vapor barrier, mylar was investigated as an adhesive to be applied by hot melting. The fusion was performed under vacuum, because mylar decomposes in the presence of oxygen at its melting temperature. Adherends were 301 stainless steel, A-110-AT titanium, and mylar film, although only stainless steel-stainless steel bonds were tested at cryogenic temperatures.

Tensile-shear strengths were measured at 20 K, RT, 422 K, and 477 K, and T-peel strengths at 77 and 295 K. Tensile-shear strengths of titanium-stainless steel and mylar-stainless steel bonds were measured at 295 K. The data show the highest tensile-shear strengths in the mylar-stainless steel bonds, lower strengths in the stainless steel-stainless steel bonds, and lowest bond strengths with titanium-stainless steel. In view of the data, the authors' conclusion that "stainless steel adherends gave the highest strength and mylar film adherends resulted in the weakest bonds" is difficult to justify. Tensile-shear strengths were highest at 295 K and lowest at 477 K, while T-peel strengths were highest at 77 K.

The authors concluded that mylar film shows promise as an adhesive for bonding mylar- or aluminized mylar-enclosed superinsulation systems to metal brackets and fuel lines, and that mylar fused bonds are useful from 20 to 422 K, but strength falls off sharply at 477 K. A severe difficulty not fully discussed by the authors is the necessity of heating under vacuum to produce the bond. In most cases, the mylar was outgassed at 625 K and bonded at 560 K. This would appear to limit the application.

HIGH TEMPERATURE THERMOPLASTIC ADHESIVES

Levine, H. H., and Trischler, F. D. (Whittaker Corp., San Diego, Calif.)
Materials and Processes for the 70's (Proc., National SAMPE Symposium
and Exhibition, 15th, Los Angeles, Calif., Apr 29-May 1, 1969) 715-8,
Society of Aerospace Material and Process Engineers (1969)

This paper contains preliminary test results on an experimental structural adhesive. The polymer was developed as a high temperature thermoplastic adhesive, but showed potential for use at cryogenic temperatures. The adhesive is not specifically identified, but resulted from modifications of completely aromatic-heterocyclic polymers to include aliphatic portions. The adhesive was applied as a hot melt tape on glass cloth carrier, bonded under pressure at 610 K for 40 seconds and cooled. Adherends were 2024 T-3 aluminum, 17-7 PH annealed stainless steel, and Ti-8-1-1 titanium alloy. Tensile-shear tests on metal-metal bonds with each adherend were conducted at 77 K, RT, 450 K, and 505 K, in some cases after aging at the higher temperatures. With aluminum and titanium, highest tensile-shear strengths were at room temperature, and strengths at 77 K were 15-25% lower. With the stainless steel, the highest bond strength was at 77 K. The lowest strengths with all adherends were at 505 K because of the thermoplastic nature of the polymer. Long-term aging, crosslinking, and fatigue tests were made at the higher temperatures. Stainless steel metal-metal bonds were made under pressure at 560 K for 30 minutes, with some loss of low-temperature bond strength. T-peel tests at 77 and 422 K gave highly erratic results, showing the need for more development, but the highest values of peel strength indicated good toughness. The authors conclude that adhesives made by modifying the structures of aromatic-heterocyclic polymers show definite potential for use from 77 to 505 K.

MEASURED EFFECTS OF VARIOUS COMBINATIONS OF NUCLEAR RADIATION, VACUUM,
AND CRYOTEMPERATURES ON AEROSPACE ADHESIVES

Lightfoot, R. P., Kerlin, E. E., and Gause, R. L. (General Dynamics/Fort
Worth, Tex.)

Effects of the Space Environment on Material (Proc., National SAMPE
Symposium and Exhibit, 11th, St. Louis, Mo., Apr 19-21, 1967) 279-90,
Society of Aerospace Material and Process Engineers (1967)

This paper summarizes work done in evaluating adhesives likely to be used in spacecraft which utilize nuclear propulsion systems. The adhesives were subjected to various environmental combinations of temperature, pressure and ionizing radiation. Tensile tests were performed on lap-shear specimens fabricated of 2024-T3 aluminum sheets bonded with the adhesives under test. The adhesives tested consisted of epoxy-nylon (4 formulations), epoxy-phenolic (3 formulations), epoxy (1 formulation), modified epoxy (1 formulation) and polyurethane (1 formulation). Specimens were irradiated in air at ambient temperatures, in vacuum, in liquid nitrogen and liquid hydrogen. Those tests termed static were done at ambient conditions after exposure to other temperatures while those tests termed dynamic were done at cryogenic temperatures without warming to room temperature. The results of this program showed that all four of these adhesive classes were quite radiation resistant and all but the modified epoxy performed well at all test conditions. Specifically, the most stable adhesives to radiation at cryogenic temperatures were the polyurethane (room temperature cure), the epoxy, and one of the epoxy-phenolics. The variations possible in formulation, cure and preparation of adhesives cause a fairly large variability in the test results. This makes quantitative comparisons difficult, but it does allow for flexibility in tailoring adhesives to particular applications.

Important references:

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2. Smith, E. T., General Dynamics/Fort Worth Report FZK-161-2, 5 Jan 1963.
3. Kerlin, E. E. and Smith, E. T., General Dynamics/Fort Worth Report FZK-188-1, 1 May 1964.
4. Kerlin, E. E. and Smith, E. T., General Dynamics/Fort Worth Report FZK-188-1, 1 May 1964.
5. Kerlin, E. E. and Smith, E. T., General Dynamics/Fort Worth Report FZK-290, 1 Jul 1966.
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RESEARCH ON POLYBENZIMIDAZOLE STRUCTURAL ADHESIVES FOR BONDING STAINLESS STEEL, BERYLLIUM, AND TITANIUM ALLOYS

Litvak, S. (Air Force Materials Lab., Wright-Patterson AFB, Ohio)
Appl. Polym. Symp., No. 3 (Structural Adhesives Bonding, Proc. of Symposium, Stevens Inst. of Tech., Hoboken, N. J., Sep 14-16, 1965), 279-98, Interscience Publishers, New York (1966)

Experimental results of tests conducted on polybenzimidazole (PBI) adhesives are reported in this paper. PBI formulations were studied over an extended period of time as it became obvious that PBI adhesives had a potentially large service temperature range. One of the larger problems associated with adhesives suitable for cryogenic temperatures has been the rapid decrease in strength above room temperature, as is the case with polyurethane. The tests reported in this paper were conducted on lap-shear structures of titanium alloys (Ti-8Al-1Mo-1V and Ti-6Al-4V), 17-7 PH stainless steel and beryllium (only a small amount of information on beryllium is given). In addition compressive-yield strength was measured for honeycomb sandwich panels of stainless steel.

The bulk of the data cover tests conducted above 273 K (tests were conducted to 800 K), but tests were run at 77 K and 20 K in the tensile-shear mode only. The results show that up to about 550 K the PBI adhesive systems exhibit substantially the same strengths as at cryogenic temperatures. Aging at higher temperatures does reduce the strength indicating that oxidation is taking place.

CRYOGENIC ADHESIVE APPLICATION

Long, R. L.

Douglas Aircraft Co., Inc., Santa Monica, Calif., Paper No. 3899.

Presented at Structural Adhesive Bonding Conference, National Aeronautics and Space Administration, Marshall Space Flight Center, Huntsville, Ala. (Mar 15-16, 1966) Paper 2, Session II, 99 pp

The paper reviews Douglas Aircraft Company experience in the selection and use of adhesives for the Saturn S-IV and S-IVB stages, in cryogenic environments. The presentation covers typical applications and corresponding requirements for adhesives, the history of the selection of hot and cold-cure adhesives, an extensive discussion of the problems with low-temperature-curing polyurethane adhesives, and some recommendations for future work.

Early in the S-IV program, a filled epoxy-phenolic was chosen as the thermosetting adhesive for large scale structures. No competitors to this adhesive had arisen to justify a change from this initial choice. An early screening of room-temperature curing adhesives led to the choice of a filled epoxy, which was particularly suited for bonding foam insulation blocks to the liquid hydrogen tank wall, and was never replaced in this application. The brittleness of this adhesive resulted in low peel strength, making it unsatisfactory for room-temperature bonding of clips and doublers. A polyurethane was chosen for this application, in spite of its disadvantages. Because of its sensitivity to water and cure cycle, and its short storage life, the polyurethane is difficult to use. The bond is subject to aging, apparently continuing to cure at room temperature, and has no useful strength at high temperatures. Attempts to find improved polymers had little success. The use of silane primers allowed an improvement in consistency of peel strengths. The addition of film materials to the bond provided some increase in bond strength, but not enough to justify the additional difficulties in processing. The quality of a polyurethane bond remained dependent on the care exercised in processing.

A number of specialized tests were devised for the polyurethane adhesive. Semimicro tests, which can be applied to very small quantities of adhesive, should be useful in programs to develop new materials.

MECHANICAL AND PHYSICAL PROPERTIES OF ORGANIC FOAMS

Lormis, F. E.

Bendix Corp., Kansas City, Mo., Final Rept. No. BDX-613-562(Rev). (Feb 1972) 60 pp. Atomic Energy Commission Contract No. AT(29-1)-613 USAEC

As part of an investigation of the properties of structural plastic foams, three adhesives were tested. The adhesives were an epoxy, a filled epoxy consisting of 40% resin and 60% aluminum oxide, and a polyurethane. Cast samples of the epoxy and the filled epoxy were tested for tensile modulus at 77 and 297 K. Thermal expansion coefficients of cast samples of all three adhesives were measured at 77, 195, 219, and 297 K. The adhesives were used to bond polystyrene foam, and the tensile properties of the bonded samples were measured to determine whether the type of adhesive caused any difference. Tensile strengths were found at 77 K and RT, and tensile modulus and elongation was measured at RT. The same tests were run on samples of polyurethane foam bonded with the three adhesives, except that tensile strength, modulus, and elongation results were all obtained at 77 K and RT. The conclusion was that little or no difference in foam tensile properties resulted from the type of adhesive used in bonding.

ADHESIVES BELOW -100 DEGREES F
Maas, M. A.
Des. News, 60-5 (Jan 6, 1965)

This brief but comprehensive review compiles technical information from a number of sources on the classes of adhesives which have been successfully tested or applied at cryogenic temperatures. The adhesives reviewed are epoxy-nylon, epoxy-phenolic, epoxy-polyamide, neoprene-phenolic, polyaromatic, polyurethane, silicone, and vinyl-acetal-phenolic. The low temperature limit of each adhesive is given as 20 K, except for the neoprene-phenolic and the silicone, given as 90 and 160 K respectively for loss of 50 percent of room temperature strength. A table compares useful temperature range, available form, cure conditions, relative cost, and advantages and disadvantages of the various classes of adhesives. The review covers each adhesive class separately, briefly discussing the properties, cure cycles, some applications, and advantages and disadvantages of the adhesives. Though brief, the review provides a good overview of the field of cryogenic adhesives in 1965.

Important references:

1. Smith, M. B. and Susman, S. E., SAE Paper 582 (1962).
2. Hertz, J., *Electro-Technology* 70, No. 3, 93-96 (Sep 1962).
3. Hertz, J., *Adhesive Age* 4, No. 8 (Aug 1961).
4. Kausen, R. C., *High- and Low-Temperature Adhesives*, Arthur D. Little, Inc.
5. Robinson, D. E. and Schreihans, F. A., *RTV Silicones as Sealants and Adhesives for Cryogenic Applications*, North American Aviation, Inc.
6. Roseland, L. M., *Evaluation of Structural Adhesives for Potential Cryogenic Use*, Douglas Aircraft Co., Inc.

OPTIMIZATION AND EVALUATION OF HIGH-TEMPERATURE STRUCTURAL ADHESIVES
Mahoney, J. W. (North American Aviation, Inc., Los Angeles, Calif.)
Air Force Materials Lab., Wright-Patterson AFB, Ohio, Tech. Rept. No.
AFML-TR-66-198, Contract No. AF 33(615)-2848 (Sep 1966) 72 pp

The objectives of this program were to evaluate potential heat-resistant organic adhesives, optimize processing, and generate engineering data on joints with the adhesives. Primary interest was in the temperature range from 530 to 650 K, but evaluations of the adhesives included temperatures as low as 20 K. Initial aging tests in the 530 to 650 K temperature range resulted in the selection of two adhesives, both polyimides, for further evaluation. Cure cycles of the adhesives were optimized for high-temperature tensile-shear strength. Adherends considered were PH14-8 stainless steel, Inconel 718, and 8-1-1 titanium, but the titanium had some bonding problems, and engineering data at low temperature were obtained with both lap bonds and honeycomb structures for PH14-8 stainless steel and Inconel 718. Tensile-shear strengths were found at 20 K, 77 K, 200 K, RT, 561 K, and 619 K; T-peel, flatwise-tension, and flexural tests were at 20 K, 77 K, 200 K, RT, 477 K, 533 K, and 644 K; and edgewise-compression testing was at 77 K, RT, 533 K, and 644 K. Additional tests not run at cryogenic temperatures were fatigue, creep rupture, salt spray exposure, humidity exposure, fluid exposure, climbing-drum peel, and creep deflection. Both adhesives showed excellent tensile-shear strengths at low temperature, but with some difference in brittleness. Both adhesives were lacking in peel strength, and results of tests on honeycomb specimens were far below values obtainable with epoxy or phenolic adhesives, but the author notes that processing was optimized for tensile-shear strength, and different cure and postcure parameters might improve other results.

ADHESIVE AND INSULATING SYSTEMS FOR CRYOGENIC AND SUPERCONDUCTING COILS
Markley, F., McKamey, A., and Biggs, J. (Argonne National Lab., Ill.)
IEEE Trans. Nucl. Sci. NS-16, No. 3, (Proc., 1969 Particle Accelerator
Conference, Accelerator Engineering and Technology, Washington, D.C.,
March 5-7, 1969), 734-5 (Jun 1969)

An organic adhesive-insulation was developed for assembly of large superconducting coils, to provide mechanical stability, interturn insulation, and sufficient flexibility to accommodate relative motions caused by temperature differentials or thermal expansion differences. The system consisted of a two-component combination of a flexible fluorinated-polymer film with very thin layers of a strong adhesive. Tests were run on a system using a fluorinated ethylene-propylene (FEP) film and epoxy adhesive combination in copper-copper bonds. Tensile-shear and block-shear tests were conducted at 77 K, with variations in FEP film thickness. Addition of the FEP film of optimum thickness increased tensile-shear strength by 25% and block shear strength by 11%, over the strengths of bonds without FEP films. Optimum thicknesses were 50 μm for block shear and 100 μm for tensile-shear results. Increased pressure during the cure cycle resulted in thinner layers of epoxy adhesive, with increased block shear strength and decreased deviation between samples at 77 K. Best surface preparation for the copper was sandblasting and acetone wash. A comparison test showed that a polyimide film gave results comparable to the FEP film at 77 K, and only slightly inferior at 300 K. Coils for a bubble chamber were constructed with an interlayer of tetrafluoroethylene resin filled with 15% glass fiber, which was easier to handle and gave results similar to the FEP film. A similar two-component bonding system, using FEP film and an epoxy adhesive, was used in bonding glass to titanium for the bubble chamber optical system. Tensile-shear strengths of glass-titanium and titanium-titanium bonds at 77 K were highest with 50 μm FEP thickness.

Important references:

1. Smith, M. and Susman, S., Development of Adhesives for Very Low Temperature, NASA-CR-52879 (May 1963).

ADHESIVES

Mauri, R. E. (Lockheed Missiles and Space Co., Sunnyvale, Calif.)
Space Materials Handbook (C. G. Goetzl, et al., Editors) 297-319,
Addison-Wesley Publ. Co., Inc. (1965)

This chapter of a handbook briefly reviews adhesives for use on aerospace vehicles, including some consideration of cryogenic-temperature service. The chapter reviews available data from the point of view of space environmental effects on adhesives, and particularly of degrading environments. The effects of high and low temperatures, nuclear radiation, high vacuum, and ultraviolet radiation, as well as some combinations of these factors, are considered. The specific cryogenic-temperature data included in the review were taken from Hertz (tensile-shear strengths of ten adhesive with four adherends, 20 to 298 K) and from Smith (effects of nuclear radiation on tensile-shear strengths of two adhesives, 20 to 297 K). Several other references, listed but not directly cited in the review, have further low-temperature information. This chapter of the handbook is not particularly comprehensive, only reviewing a few examples from the available data. In the third edition of the handbook, (see abstract on page 81), published three years later, the author has completely rewritten the chapter, in a longer and more comprehensive form. The two versions review completely different material; in fact, not even one reference appears in the reference lists of both versions.

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2. Smith, E. T., Report FZK-616-2, General Dynamics Corp., Fort Worth, Texas (Jan 1963).
3. Hiza, M. J. and Barrick, P. L., Society of Plastics Engineers Transactions 1, No. 2, 73-79 (Apr 1961).
4. Hertz, J., Report ERR-AN-032, ASTIA AD 273219, General Dynamics Corp., Convair Astronautics, (25 Jan 1961).
5. Miller, R. N., et al., Ind. Eng. Chem. Product Research and Development 1, No. 4, 257-261 (Dec 1962).
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7. Smith, M. B. and Susman, S. E., Report 582C, Society of Automotive Engineers (Oct 1962).

ADHESIVES

Mauri, R. E. (Lockheed Missiles and Space Co., Palo Alto, Calif.)
Space Materials Handbook, Third Edition (J. B. Rittenhouse and J. B. Singletary, Editors) 241-92, National Aeronautics and Space Administration Spec. Publ. NAS-SP-3051, Air Force Materials Lab., Wright-Patterson AFB, Ohio, Technical Report AFML-TR-68-205 (1968)

This chapter of a handbook reviews data on structural adhesives used in aerospace technology. The review summarizes the available data on the separate and combined effects of elevated temperature, cryogenic temperature, high energy particulate and electromagnetic radiation, and vacuum on the more important classes of adhesives. A section on nonstructural (transparent) adhesives and a 17-reference bibliography complete the review. Specific data at cryogenic temperatures used in the review were taken from Kuno (sixteen classes of adhesives including ten with service temperature ranges to 20 K; tensile-shear strengths of four adhesives, stainless steel adherends, 20 to 800 K; tensile-shear strengths of five adhesives, aluminum adherends, 20 to 470 K), from Smith and Susman (tensile-shear strengths of 13 adhesives on aluminum adherends, 20 to 355 K; peel strengths of 13 adhesives on aluminum adherends, 20 to 355 K; linear expansions of eight adhesives between 77 and 273 K), from Roseland (fatigue behavior of six adhesives at 77 and 297 K; compatibility of polyurethane adhesive with liquid hydrogen and liquid oxygen), from Gray, et al. (effects of vacuum and radiation on tensile-shear strengths of five adhesives at 77 K), and from Lightfoot and Kerlin (effects of gamma radiation on tensile-shear strengths of nine adhesives, 20 to 297 K). This chapter is quite comprehensive in summarizing the space environmental effects on adhesives. A previous version of this chapter (see abstract on page 80) is so different that it must be considered as a separate paper.

Important references:

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2. Smith, M. B. and Susman, S. E., NASA Final Summary Report No. CR 52879, May 1963.
3. Roseland, L. M., Seventh Annual SAMPE National Symposium Transactions, Los Angeles, Calif., 20-22 May 1964, pp 7-1 to 7-17.
4. Kerlin, E. E. and Smith, E. T., Report No. FZK-172, General Dynamics, Fort Worth, TX (30 Sep 1963); and Report No. FZK-290 (1 Jul 1966).
5. Gray, P. D., et al., Report No. RTD-TDR-63-1050, Aerojet General Corporation (Feb 1963).
6. Lightfoot, R. P. and Kerlin, E. E., Eleventh Annual SAMPE National Symposium Transactions, St. Louis, Missouri (10-21 Apr 1967), pp 279-290.

EPOXY RESINS AS CRYOGENIC STRUCTURAL ADHESIVES

McClintock, R. M., and Hiza, M. J. (National Bureau of Standards, Boulder, Colo. Cryogenic Engineering Lab.)

Adv. Cryog. Eng. 3 (Proc., Cryogenic Engineering Conference, Boulder, Colo., August 19-21, 1957), 305-15, K. D. Timmerhaus, Ed., Plenum Press, Inc., N.Y. (1960)

Filled epoxy resins were investigated as adhesives for copper-copper bonds, to provide design information for a liquid hydrogen cooled electromagnet. The epoxy resin paste consisted of about 45% resin and 55% an inorganic filler material, predominantly alumina and asbestos. The curing agent was diethylamino-propylamine. Experimental data were collected on the impact strength of the cured adhesive in bulk at 76 and 300 K; thermal contraction of the cured adhesive between 300 and 76 K; tensile and shear strengths of copper-copper bonds at 20, 76, and 300 K; and the effects on tensile and shear strengths of exposure to high and low temperatures, and of adhesive thickness. Impact strengths were low at room temperature and even lower at 76 K. Tensile strengths were not affected by test temperature and only slightly affected by rapid thermal cycling between 76 K and room temperature before testing. Shear strengths were lower at 20 and 76 K than at room temperature, and were decreased by thermal cycling before testing. Exposure to temperatures above the cure temperature seriously degraded tensile and shear strengths. Variation of adhesive thickness between 250 and 750 μm had no effect on strength. The authors conclude that thermal stresses are a primary cause of strength loss in adhesive bonds at cryogenic temperatures. The sources of such stresses, including cure shrinkage and differential thermal contraction between adhesive and adherend, are discussed.

EFFECTS OF NUCLEAR RADIATION AND CRYOGENIC TEMPERATURES ON NONMETALLIC ENGINEERING MATERIALS

McKannan, E. C., and Gause, R. L. (National Aeronautics and Space Administration, Marshall Space Flight Center, Huntsville, Ala.)
J. Spacecr. Rockets 2, No. 4, 558-64 (Jul-Aug 1965)

This paper summarizes the work performed under contract to NASA by General Dynamics Corporation on the testing of nonmetallic materials at various combinations of radiation levels and temperatures (ambient and 77 K). The material classes included 1) structural adhesives, 2) structural laminates, 3) thermal insulations, 4) electrical insulations, 5) potting compounds, 6) seals, 7) sealants, 8) lubricants and 9) thermal control coatings. This paper discusses the results on classes 1) through 4) above.

Results are presented for two adhesives, a polyurethane type and an epoxy-nylon type. The tests were conducted at 323 K, 77 K and 20 K with radiation doses ranging from zero to 6×10^{10} ergs/gm. The test arrangement was such that the tensile-shear tests which followed irradiation were conducted without changing the temperature following the irradiation. The results for the polyurethane showed a substantial increase in strength as the temperature is lowered as well as an increase after ambient irradiation. The high strength exhibited by this adhesive at cryogenic temperatures was degraded somewhat by the radiation exposure, but the strengths were always substantially above the ambient, control values. The epoxy-nylon adhesive exhibited a substantial reduction in strength as the temperature was lowered as well as further reduction after irradiation. The authors interpret these reduced values for the shear strength as a result of temperature and radiation induced embrittlement. This adhesive was judged to be unacceptable for use above an exposure of 10^{10} ergs/gm under ambient conditions and unacceptable at cryogenic temperatures with no radiation exposure. The latter results are corroborated by other tests reported in the literature.

Important references:

1. Lucas, W. R., Symposium on Space Radiation Effects, Publ. 363, American Society for Testing Materials, Philadelphia, Pa. (1964).
2. Smith, E. T., 1963 Summer General Meeting of the Institute of Electrical and Electronics Engineers, Toronto, Canada (1963).
3. Lockheed Missiles and Space Co., Contract NAS8-5600, Rept. NSP-63-35 (May 1963).
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5. Smith, M. B. and Susman, S. E., NARMCO Research and Development Co., Final Summary Report, NASA Contract NAS8-1565 (May 1963).
6. Smith, E. T., Annual Report, Volume II, NASA Contract NAS8-2450 (Nov 1961 thru Nov 1962).

ADHESIVES

McKillip, W. J. (Ashland Chemical Co., Bloomington, Ind.)

J. Br. Interplanet. Soc. 24, No. 11, 659-84 (Nov 1971)

This paper is a comprehensive review of adhesives for use in missiles and space vehicles. After a summary of the applications for cryogenic adhesives and the causes of stress concentrations in adhesive joints, the cryogenic adhesives are categorized by chemical types and properties. The epoxy type adhesives are discussed rather briefly, and the author concludes that they have been inadequate as cryogenic adhesives for space application. Following this is a much more extensive discussion of the urethane-type adhesives, which are said to hold most promise as structural adhesives for cryogenic applications. The discussion includes tables of data taken from Roseland (see abstract on page 110), comparing the properties of modified epoxies and polyamide epoxies to polyurethane adhesives (tensile-shear and T-peel strengths of seven adhesives at 23 and 393 K; tensile-shear and T-peel strengths of three adhesives at six temperatures from 23 to 393 K; tensile strengths of insulation foam sandwiches using three adhesives at six temperatures from 23 to 393 K; and fatigue strengths of three adhesives at 78 K and RT). A brief discussion of silicone adhesives notes that RTV silicones are adequate for non-structural bonds at cryogenic temperatures, and withstand exposure to higher temperatures than the polyurethanes can tolerate. Another brief discussion of fluorocarbon hot melt adhesives notes LOX compatibility. The remainder of the paper deals with adhesives at high temperatures, although the discussion on polybenzimidazole adhesives includes some data from Litvak (see abstract on page 74) at cryogenic temperatures (tensile-shear strengths at 23 and 78 K). The paper concludes with 40 references, of which 16 are related to cryogenic adhesives.

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12. Lantz, R., 6th National SAMPE Symposium, Materials for Space Vehicle Use, Seattle (Nov 1963).
13. Hertz, J., MRG-95 (25 Aug 1959).
14. Hill, W., Structural Bonding Symposium, NASA Space Flight Center, Huntsville, Ala. (15 Mar 1966).
15. Schreihans, F., and Robinson, D., Proceedings 7th National Symposium on Adhesives and Elastomers, SAMPE, Los Angeles (May 1964) Section 6.
16. Litvak, S., Technical Report AFML-TR-65-426 (Mar 1966).

EVALUATION OF CRYOGENIC INSULATION MATERIALS AND COMPOSITES FOR USE IN
NUCLEAR RADIATION ENVIRONMENTS. MATERIALS TESTS

McMillan, W. D., Bradbury, H. G., Carter, H. G., Lightfoot, R. P., and
Kerlin, E. E.

General Dynamics/Fort Worth, Tex., Rept. No. FZK-347, National Aeronautics
and Space Administration Rept. No. NASA-CR-61920, Contract No. NAS8-18024
(May 1968) 264 pp

The objective of this program was to evaluate cryogenic insulation materials for application to a nuclear rocket vehicle, where the materials are exposed to cryogenic temperatures and nuclear radiation. Four adhesives, two polyurethanes and two epoxy-polyamides, were among the materials tested. Aluminum-aluminum bonds with each adhesive were subjected to gamma and neutron irradiation in air at RT or in liquid hydrogen at 20 K, and tested for tensile-shear or T-peel strengths at RT. A few samples were irradiated at 20 K and tested for tensile-shear strength at 20 K. Room-temperature tensile-shear strengths of the epoxy-polyamides were higher than those of the polyurethanes before and after all irradiations. In tests at 20 K, one of the polyurethanes had a much higher tensile-shear strength before irradiation than the other three adhesives, which were nearly equal to each other. The polyurethanes suffered the greatest decrease after irradiation, with the tensile-shear strength of the best one brought down to the level of the two epoxy-polyamides. In most cases, irradiation decreased the tensile-shear strengths. Exceptions were the epoxy-polyamides irradiated and tested in liquid hydrogen, which showed no change, and one of the polyurethanes irradiated in liquid hydrogen and tested at RT, which actually gained in strength. Irradiations in air caused greater strength losses than irradiations in liquid hydrogen, but this was attributed to the larger radiation doses achieved in air. Peel strengths were more variable. Irradiation in air caused losses of peel strength in all four adhesives, but one polyurethane and one epoxy-polyamide showed increases in peel strength with irradiation in liquid hydrogen, while the other adhesives lost strength.

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THERMAL CONDUCTIVITY OF GENERAL ELECTRIC NO. 7031 VARNISH
McTaggart, J. H., and Slack, G. A. (General Electric Co., Schenectady,
N.Y.)
Cryogenics 9, No. 5, 384-5 (Oct 1969)

An insulating varnish which has been used as an adhesive at cryogenic temperatures was tested. The varnish air dries to a pliable resin, which can be baked as a thermoset adhesive. The varnish is identified as a vinyl modified phenolic. A rod shaped sample was prepared and baked, and its thermal conductivity was measured between 2.5 and 300 K. The results show a thermal conductivity comparable to glass or other plastics commonly used at low temperatures, confirming the usefulness of the varnish as a cryogenic temperature adhesive.

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1. Ashworth, T. and Rechowicz, M., Cryogenics 8, 361 (1968).

PROPERTIES OF FOAMS, ADHESIVES, AND PLASTIC FILMS AT CRYOGENIC TEMPERATURES

Miller, R. N., Bailey, C. D., Freeman, S. M., Beall, R. T., and Coxe, E. F. (Lockheed-Georgia Co., Marietta)

Ind. Eng. Chem., Prod. Res. Dev. 1, No. 4, 257-61 (Dec 1962)

The results reported are from a program to develop a foam insulation system for the liquid hydrogen tanks of space vehicles. The insulation system consists of foam, vapor barrier film and the necessary adhesive to bond the materials to each other and the stainless steel or aluminum tank. The tests conducted on the adhesives consisted of shear-tensile tests with two strips of stainless steel or aluminum lap bonded to the same piece of vapor barrier material. The adhesives tested were an epoxy, three epoxy-polyamides and a commercially available adhesive. Tests were conducted at 20 K and 296 K. The vapor barrier materials were polyethylene terephthalate, polyvinylfluoride and fluorohalocarbon film. The foam insulations investigated included polyurethane, polyethylene, polyether, cork and neoprene sponge. The metals were either 302 stainless steel or 5034 aluminum alloy. The results showed that the epoxy and one of the epoxy-polyamides had the greatest strength at both temperatures, however the epoxy-polyamide had an advantage as it cures at room temperature. The vapor barrier materials were tested for ultimate tensile strength and elongation at 77 K and the foams were tested under compression load at 77 K and 296 K.

The final component selection consisted of an epoxy-polyamide adhesive, polyethylene terephthalate vapor barrier and polyether foam.

Important references:

1. Haskins, J. F. and Hertz, J., *Advan. Cryo. Eng.* 7, 353-9 (1962).
2. Hertz, J., *Advan. Cryo. Eng.* 7, 336-42 (1962).
3. Hertz, J., *ASTIA Rept. AD 273219* (Jan 25, 1961).
4. Smith, M. B. and Susman, S. E., *Narmco Research and Development, Paper E-8, Cryogenic Engineering Conference, Aug 1962.*

WHICH LOW TEMPERATURE ADHESIVE IS BEST FOR YOU?

Miska, K. H.

Mater. Eng. 81, No. 5, 52-4 (May 1975)

This paper concisely reviews the adhesives available for applications at temperatures below about 250 K. The first class of materials noted is urethane adhesives, which have the best properties at temperatures as low as 20 K. Disadvantages of the urethanes include relatively low strength at 298 K, bond degradation due to moisture, and toxicity of the components. The silicone adhesives are said to be useful over a range from 200 to 533 K, as sealants or in joints designed to take advantage of the relatively high peel strengths. Tensile and tensile-shear strengths are quite low. Room temperature vulcanizing silicone adhesives and sealants and phenylmethyl silicones claim useful properties as low as 111 K. The epoxy formulations are ranked in order from epoxy-nylon, the best, through epoxy-polysulfide, epoxy-phenolic, epoxy-polyamide, amine-cured, and anhydride-cured epoxy, based on tensile-shear strengths below 218 K. An experimental amine-cured urethane-modified epoxy is said to show high tensile strength and good elongation at 5 K. Some modified acrylics are useful as adhesives down to 200 K, and some rubber-based adhesives can be used for sealing or bonding as low as 210 K. A tabulation gives average tensile-shear strengths of epoxy-nylon, epoxy-phenolic, and polyurethane adhesives from 20 to 298 K, epoxy-polysulfides down to 116 K, silicones to 218 K, and modified acrylics and an experimental copolyester polymer to 233 K. Four specifications applicable to low temperature adhesives are listed. The paper does not give specific sources for the properties of the adhesives. The references listed in the paper are general references on adhesive bonding techniques, rather than low temperature applications.

SUPERFLUID HELIUM CONTAINER OF MYLAR CONSTRUCTION

Moss, T. H., Kellers, C. F., and Bearden, A. J. (Cornell Univ., Ithaca, N.Y. Lab. of Atomic and Solid State Physics)
Rev. Sci. Instrum. 34, No. 11, 1267-8 (Nov 1963)

This note briefly describes the construction of a superfluid helium container constructed of Mylar with adhesive bonding of all joints. The adhesive used was an epoxy, selected after "considerable experimentation" to find the epoxy adhesive with the highest "peel strength" with Mylar. A Mylar sheet was rolled into a tube with the seam adhesively bonded, Mylar end windows and aluminum stiffener rings were bonded to an end of the tube and to a brass fitting, and the tube was bonded to the brass fitting. The same epoxy adhesive was used for all bonds, including mylar-mylar, mylar-aluminum, and mylar-brass. The assembly achieved containment of superfluid helium, and epoxy-bonded mylar structures showed promise for other cryogenic applications.

Important references:

1. Mehr, D. L. and McLaughlin, E. F., Rev. Sci. Instr. 34, 104 (1963).

A CONVENIENT AND RELIABLE DEMOUNTABLE SEAL FOR LOW TEMPERATURE WORK
Mota, A. C. (California Univ., San Diego. Dept. of Physics and Inst.
for Pure and Applied Physical Sciences)
Rev. Sci. Instrum. 42, No. 10, 1541-2 (Oct 1971)

This note briefly describes a combination of sealant and seal design which made a reliable leak-tight seal against superfluid or liquid helium. The sealant was a solution of soap flakes in glycerine. The seal design necessary for reliable leak-tightness used a carefully machined threaded connection, with the sealant applied to the last threads and flat faces of the connecting pieces. Epoxy-epoxy, copper-copper, and brass-brass joints were successfully sealed by the technique. As long as precautions were taken to avoid sudden temperature changes, seals remained leak-tight through several cycles between room temperature and liquid helium temperature. The author warns that departures from the specified seal design can lead to failure.

PROPERTIES OF NONMETALLIC MATERIALS AT CRYOGENIC TEMPERATURES

Mowers, R. (Rocketdyne, Canoga Park, California)

Proceedings of the 1968 Summer Study on Superconducting Devices and Accelerators, Part I, Brookhaven National Lab., Upton, N.Y, Rept. No. BNL 50155 (C-55) 326-67 (Apr 1969)

This paper reviews structural applications for non-metallic materials at cryogenic temperatures. The applications reviewed are separated into five classifications, one of which is adhesives, sealants, and coatings. Within each classification, reports of representative test programs are described in terms of title, source, objectives, materials tested, properties measured, data obtained, and conclusions drawn. Some tables and graphs are extracted from reports as examples of the data available.

Three reports are summarized for adhesives and sealants. First of these is by Smith and Susman (see abstract on page 124), on a program to evaluate commercially available adhesives for cryogenic application, which found a nylon powder-filled epoxy-polyamide paste the best system, and a polyurethane elastomer adhesive good at low temperatures but tending to produce unsatisfactory joints because of absorption of moisture from air. A graph from the report shows tensile-shear strengths at 77 K of 19 adhesives. The second report summarized is by Kuno (see abstract on page 65), from the same organization as the first report, but with later data, which concluded that polyurethanes were very good at extremely low temperatures but lost strength as temperature increased toward ambient, while nylon-epoxy materials had rather uniform strength from 20 to 373 K. Graphs extracted from the paper show tensile-shear strengths of 5 adhesives from 20 to 350 K and tensile-shear strength of 8 adhesives at 20 K. The third report summarized is by Akawie (see abstract on page 3), on sealants for sealing or repairing cryogenic fluid containers. Eight polymer systems were investigated and only reinforced silicones and polyurethanes met requirements at 77 K.

BORON/EPOXY SUPPORT STRUT FOR NON-INTEGRAL CRYOGENIC TANKAGE

Nadler, M. A., Yoshino, S. Y., and Darms, F. J., Jr. (North American Rockwell Corp., Downey, Calif. Space Division)
Materials and Processes for the 70's (Proc., National SAMPE Symposium and Exhibition, 15th, Los Angeles, Calif., Apr 29-May 1, 1969), 179-207, Society of Aerospace Material and Process Engineers (1969)

This paper deals with the design and development of a prototype boron-epoxy composite strut to support the liquid hydrogen tank in a future nuclear space flight stage. The cylindrical body of the strut is made up of several concentric layers of longitudinally oriented boron fibers in an epoxy matrix. At the ends of the strut, the layers are separated and interleaved with metal shims, adhesively bonded to the layers, for attachment to end fittings. A screening program evaluated six adhesives with four metallic adherends, using four primers or no primer, and with two surface preparations. The adhesives and primers are identified by their commercial designations. Tensile-shear strengths at 89 K and RT were used to evaluate systems. Not all of the test results are shown, but final results on the boron composite - 18% nickel maraging steel bonds, using the best adhesive with or without the best primer, are tabulated. Strengths at 89 K were less than half those at RT, and the primer increased bond strength slightly. Simulated shim joints were made with AM-355 steel shims and loaded to failure at 89 K and RT. Partial test results are given, in which the failing loads did not quite meet the minimum load capacity expected of the boron composite. The authors outlined further testing to be done, and concluded that improved designs had been conceived for joint fittings of circular tubes.

Important references:

1. Smith, M. B. and Susman, S. E., Development of Adhesives for Very Low Temperature Applications, Final Summary Report NASA CR-52879 (May 1963).

EPOXY RESIN AS A MATERIAL FOR CONSTRUCTING CRYOGENIC APPARATUS
Netzel, R. G., and Dillinger, J. R. (Wisconsin Univ., Madison. Dept.
of Physics)
Rev. Sci. Instrum. 32, No. 7, 855 (Jul 1961)

An epoxy resin was successfully used as both a construction material and a cement for apparatus for use at temperatures below 1 K. The epoxy was cast in brass or Lucite molds, and machined to desired shapes including capsules. As cement, the epoxy was used to seal epoxy covers, electrical leads, and stainless steel pipes to the epoxy capsules. Assembled capsules were cooled repeatedly from room temperature to temperatures below 0.1 K with no failures. Epoxy-epoxy, epoxy-stainless steel, and glass-glass seals made with the epoxy resin were found to be vacuum tight in the presence of superfluid helium, demonstrating the extremely good bonds formed with the epoxy resin.

ADHESIVES - CRYOGENIC FOR USE WITH TEMPERATURE TRANSDUCERS

Nirschl, D. A.

General Dynamics/Convair, San Diego, Calif., Rept. No. GDC-ZZC-64-017
(Feb 1964) 41 pp

Tests were performed to select a suitable commercial adhesive for bonding temperature transducers to stainless steel cryogenic tanks of space vehicles. The adhesives considered were 18 commercial adhesives, identified in the report only by their commercial designations. The temperature transducers were of two types, identified as platinum-germanium transducers and mylar transducers. Except in the time response and vacuum tests, these transducers were simulated by platinum blocks and mylar films bonded to stainless steel. Temperature shock tests were run on platinum blocks and mylar films bonded to the outside bottom of a container, by filling the container with liquid nitrogen, then returning to room temperature, for five temperature cycles. The mylar films were "peeled" off with pliers at room temperature or 200 K, and the platinum blocks were removed by rocking with pliers at room temperature, the bond was examined microscopically, and the adhesives were rated for relative bond strengths. The chip test consisted of cooling mylar film samples to 77 K as before, returning to 200 K, chipping the squeezed-out bondline with a chisel, and rating the bond as hard or brittle. Application comparison tests rated the relative "peel" strengths of mylar film samples with variations in surface preparation. Best results were with sanded and heated surfaces.

These screening tests eliminated 12 of the 18 adhesives from further consideration. The remaining adhesives were subjected to environmental tests. Temperature shock tests consisted of five cycles of rapid cooling to 20 K and warming to room temperature, followed by five cycles between 20 and 333 K. For impact tests, the test fixture was impacted with a steel ball at 20 K. After these environmental tests, bond strengths were evaluated as before by peeling or rocking with pliers. Time-response was tested with the actual transducers, bonded to stainless steel. The platinum-germanium samples were dipped into liquid hydrogen, and their response times recorded. The Mylar film transducers were tested the same way in liquid nitrogen. The final test evaluated relative bond strengths with the bonded transducers after exposure to vacuum. Two of the adhesives were recommended for use in space vehicles.

ADHESIVE PROPERTIES -300 TO 700F

North American Aviation, Inc.

North American Aviation Inc., Downey, Calif., Rept. No. AL-2590, Contract No. AF 33(600)-28469 (Sep 1957) 40 pp

Purpose of the experimental program was to evaluate selected adhesives for use in structural joining at temperatures between 89 and 644 K, for periods up to 5 hours. The adhesives tested in the program included an epoxy-phenolic, an epoxy, an epoxy-polyamide, two modified phenolics, and two systems identified by their commercial designations. Adherends were aluminum honeycomb core and aluminum face sheets. Sheet-sheet lap joints were tested for tensile-shear strengths, and core-sheet sandwich structures were tested for peel strength, and as two-point loaded beams, loaded to make either skin compression or core shear the critical factor. All tests were run at 89 K, 218 K, and RT to establish low-temperature behavior. Other tests with glass-reinforced plastic and stainless steel adherends were run at temperatures of 561 and 644 K. The two adhesive systems identified by their commercial designations were superior at low temperatures, one in the tensile-shear tests and the other in the peel tests. The sandwich beam tests showed that any of the adhesives could stabilize the sandwich structure at the design stress levels. A 5-hour soak at 89 K had no apparent effect on the ability to stabilize the structure. In the high temperature tests, the epoxy-phenolic and modified phenolics achieved significant tensile-shear strengths even at 644 K for 5 hours exposure.

LEFKOWELD 109/LM-52 AND NARMCO 7343/7139 BOND STRENGTH TO 3D FOAM AT 25°C AND -196°C

Oliensis, G. M., and Rumenapp, H. E.

Douglas Aircraft Co., Inc., Santa Monica, Calif. Missile and Space Systems Div., Rept. No. SM-48414 (Aug 1965) 73 pp

In assembling the liquid hydrogen tanks of the Saturn S-IVB, a three-dimensionally oriented glass fiber reinforced polyurethane foam is adhesive-bonded to the aluminum wall, then covered with a fiberglass-cloth composite protective liner laminated in position. Sample coupons are manufactured and cured under the same conditions at the same time, and tests on the coupons are used to determine acceptability of the bonding operation in the tank. Tensile tests are run on the coupons at 77 K, a more practical and less expensive process than testing at 20 K, but a great deal of trouble and expense could be saved if the low-temperature strength could be predicted from room-temperature tests. This program was conducted to establish such a correlation, and it also provided experimental data on adhesive bonds at cryogenic temperatures.

The adhesives involved were a filled modified epoxy (aluminum-foam bond) and a polyurethane (foam-liner bond). Samples were tested for butt-tensile strength of the bond at 77 K and room temperature. Laboratory samples were made with a variety of manufacturing process variables, to find the temperature correlation and the effect on it of the process variables. Test results on the epoxy adhesive showed no failures (all samples had satisfactory strength at 77 K), in contrast to quality-control experience in tank manufacture. No correlation with room temperature results was possible with no failures, and it was apparent that the actual controlling variables were not being tested. The situation was different for the polyurethane, where a correlation was found between strengths at room and cryogenic temperatures. However, because of wide variability in test results, use of the correlation could lead to an excessive rejection rate. It was found that appearance of low resin content in the liner was an unreliable means of predicting weak bond strength.

INVESTIGATION OF STRUCTURAL ADHESIVES FOR CRYOGENIC APPLICATIONS

Pascuzzi, B., and Hill, J. R. (Boeing Co., Seattle, Wash.)
Plastics and Adhesives in the Space Age (Regional Technical Conference,
Garden City, N.Y., May 13-14, 1964) 87-103, R. D. Forger, Ed., Society
of Plastics Engineers, Inc., N.Y. (1964)

Objective of the work was to develop a simple adhesive bonding process for 2219 aluminum, for attaching various fittings to the external surface of a rocket engine case. The application requires simplicity of processing and bond strength reliability under static and dynamic loading at temperatures from 77 to 366 K. Four adhesives and two metal surface preparations were investigated initially. The adhesives were an epoxy-polyamide, a polyurethane, and two glass fabric supported epoxies. The aluminum surface preparations were abrasion and a chemical treatment. Initial selection was based on tensile-shear tests at 77, 297, and 366 K and vibration-fatigue tests at 77 K. In all but two combinations of adhesive and surface preparation, highest tensile-shear strength was observed at room temperature. The polyurethane with chemically treated aluminum and one epoxy with abraded aluminum showed tensile-shear strength increasing with each temperature decrease. The strengths of these two combinations at 77 K were nearly equal, and far superior to the other systems.

Vibration-fatigue tests showed a clear superiority of the polyurethane over the other adhesives. The combination of polyurethane adhesive with chemically-treated aluminum was selected for further testing, including tensile-shear after thermal cycling between 77 and 366 K, vibration at 77 K and thermal shock to 77 K under load with shear loading, shear impact, T-peel, and flatwise tensile at temperatures from 77 to 366 K, liquid oxygen impact compatibility of an assembled aluminum-aluminum bond, and humidity and salt spray exposure. The system showed marginal strength properties at 77 K in T-peel tests, and showed peel-type failures under the shear loading used in vibration and thermal shock tests at 77 K. Marginal strength properties at 297 and 366 K were observed under impact and following environmental exposure.

The system is considered by the authors to be suitable for structural applications involving temperatures down to 77 K, as long as the structure design avoids critical peel and bending loads.

This paper is also published with very slight variations in Adhesives Age (see citation in Secondary Documents).

ADHESIVES, SEALANTS, AND GASKETS--A SURVEY

Perkins, R. B., and Glarum, S. N. (Southern Research Inst., Birmingham, Ala.)

National Aeronautics and Space Administration Spec. Publ. NASA-SP-5066
(1967) 63 pp

This survey is part of the technology utilization program, and reviews contributions made by NASA or under NASA sponsorship to the technology of adhesives, sealants and gaskets. The survey considers each major program separately. The first one considered is a two-year program on adhesives for very-low-temperature application. This program identified nylon-epoxy, epoxy-polyamide, and polyurethane adhesives as having the most promise, then developed and evaluated six new adhesives specifically designed for use at low temperatures. These new adhesives were a nylon-filled epoxy-polyamide, an epoxy-polyamide with two teflon films included in the bond line, three polyurethanes including one supported on glass fabric, and a teflon sheet used as a hot-melt adhesive. Tensile-shear, T-peel, impact, and butt-tensile tests at temperatures from 20 to 355 K were used to evaluate the adhesives, and the effectiveness of fillers, supports, and flexible films included in the bond line was demonstrated. Much of the program was concerned with studies of nylons in adhesives. Epoxies, phenolics, acrylics, polyesters and polyurethanes were compared as reinforcing agents for nylon adhesives. Various modified nylons were formulated and evaluated as adhesives and as additives to epoxy adhesives. Nylon was compared to inorganic powders as filler for an epoxy-polyamide adhesive.

Another program described in the survey developed a method of attaching aluminum studs to an aluminum structure, using a polyurethane-modified epoxy room-temperature curing adhesive with a flexible polyurethane film included in the bond line.

A third program was concerned with developing adhesives that would perform satisfactorily over a temperature range from 20 to 478 K. Potentially useful new types of polymers were developed, although no attempt was made to develop finished adhesives. The polymer classes investigated were polyisocyanates, epoxies, epoxy esters, poly-2-oxazolidones, polyimides, and polyisocyanurates.

A fourth program involved development of elastomeric sealants for use at temperatures as low as 20 K. Commercial polymers were evaluated for flexibility in bend tests at 77 K, and while all of the polymers cracked, one polyurethane and one silicone were judged superior for use as low temperature sealants. Thermal contraction tests between 265 and 77 K showed that the polyurethane had less thermal contraction, which

led to its choice for further experiments. Filling the polyurethane sealant with glass fibers decreased the thermal contraction by two-thirds, and tests of filled polyurethane sealants showed no visible damage caused by thermal shock cycling between 77 K and room temperature. Investigations were continuing on the synthesis of new polymers for application as sealants.

The survey is a comprehensive summary of all of these programs, and refers to other reports for more detailed information.

Important references:

1. Kuno, J. K., Comparison of Adhesive Classes for Structural Bonding at Ultra-High and Cryogenic Temperature Extremes, Seventh Nat. Symp., Soc. Aerospace Mater. and Process Eng. (May 1964).
2. Kausen, R. C., High- and Low-Temperature Adhesives--Where Do We Stand. Seventh Nat. Symp., Soc. Aerospace Mater. and Process Eng. (May 1964).
3. Roseland, L. M., Evaluation of Structural Adhesives for Potential Cryogenic Usage, Seventh Nat. Symp., Soc. Aerospace Mater. and Process Eng. (May 1964).
4. Smith, M. B. and Susman, S. E., Summary Rept., Narmco Research and Development (NASA Contract NAS8-1565), Jul 15, 1962.
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Important references (continued):

13. Sandler, S. R., Berg, F. and Kitazawa, G., Monthly Rept., The Borden Chemical Co. (NASA Contract NAS8-11518), Jan 1965.
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18. Hiza, H. M. and Barrick, SPE Trans. 1, 93 (Apr 1961).
19. Zelman, I. M., Akawie, R. I. and Bahun, C. J., Final Rept., Hughes Aircraft Co. (NASA Contract NAS8-2428), 1962.
20. Zelman, I. M., Akawie, R. I. and Harvey, H., Summary Rept., Hughes Aircraft Co. (NASA Contract NAS8-2428), 1963.
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CEMENTING TEFLON AT LOW TEMPERATURES

Reichert, J. F. (State Univ., of New York, Buffalo. Physics Dept.)
Rev. Sci. Instrum. 43, No. 11, 1727-8 (Nov 1972)

This brief note describes an adhesive which has provided strong, reliable low-temperature bonds between teflon and various metals, plastics, and glass. The adhesive is ordinary rubber cement, applied to both surfaces to be joined, partially dried for about a minute before the pieces are joined, and the assembly dried for about 10 minutes before cooling, although the entire procedure is not critical. The note provides no specific experimental data, but the bond is said to become stronger at liquid helium temperature and to remain strong after repeated cycling between room and liquid helium temperatures. Other rubber base adhesives fail to hold for a single temperature cycle. The author concludes that ordinary rubber cement is an easy to use, cheap, readily removable, non-destructive, strong at low temperature, and reliable adhesive for teflon.

MECHANICAL PROPERTIES OF IMIDITE ADHESIVES

Reinhart, T. J., Jr., and Hidde, R. (Air Force Materials Lab., Wright-Patterson AFB, Ohio)

Appl. Polym. Symp. No. 3 (Structural Adhesives Bonding, Proc. of Symposium, Stevens Inst. of Tech., Hoboken, N.J., Sep 14-16, 1965), 299-318, Interscience Publishers, New York (1966)

Mechanical property tests were conducted on lap-shear and honeycomb-core sandwich panels using polybenzimidazole (PBI) resin systems as adhesives. Tests were conducted at 20 K, 77 K, 200 K and up to 750 K. The tests included lap-shear tensile tests (down to 20 K), fatigue tests (down to 77 K), creep rupture tests (all at room temperature and above), T-peel tests (to 20 K), sandwich-peel tests (to 77 K), sandwich panel flatwise tension tests (to 77 K), sandwich panel shear tests (to 77 K), sandwich beam flexure tests (to 77 K) and sandwich panel edgewise compression tests (to 77 K). The bulk of the tests were conducted on 15-7 Mo stainless steel, but lap-shear and fatigue tests were done using 2219 aluminum alloy and Ti-6Al-4V. Some of the tests were conducted after aging for up to 1000 hours at the test temperature.

The bonds investigated exhibited good thermal stability for long exposures from cryogenic to moderately elevated temperatures. Long exposure at higher temperatures (greater than 600 K) resulted in failures during heat aging. The adhesive system exhibited no creep. The stainless steel and titanium specimens had fewer failures at low temperature than the aluminum. The peel strength at elevated temperatures was higher than at room temperature and below. The only adverse result of the cryogenic tests was a slight decrease in strength in the beam flexure tests.

Important references:

1. Hidde, R. and Reed, R., Whittaker Corporation, Summary Technical Report, Feb 1965, Contract No. AF 33(615)-1207, San Diego, Calif.

APPLICATION OF AN ADHESIVELY BONDED CRYOGENIC INSULATION SYSTEM

Rittenhouse, J. B. (Lockheed Missiles and Space Co., Palo Alto, Calif. Materials Sciences Lab.) Structural Adhesive Bonding Conference, National Aeronautics and Space Administration, Marshall Space Flight Center, Huntsville, Ala. (Mar 15-16, 1966) Paper 1, Session III, 25 pp

This paper reports on a program to develop a cryogenic insulation system for the Saturn V Apollo liquid hydrogen vessel. The system developed was an adhesive bonded multilayer insulation over a helium-purged fiberglass substrate. An earlier liquid hydrogen tank had an internal insulation consisting of polyurethane foam bonded to the tank wall with an epoxy adhesive, and coated with a fiberglass-epoxy resin seal coat. This insulation had withstood several fillings with liquid hydrogen without failure. The same epoxy adhesive was used to bond Velcro fasteners to an aluminum tank outer surface, to attach the purge bag for the new insulation. A screening program was carried out to find the best adhesive to attach the Velcro fasteners to the fluorohalocarbon polymer film purge bag. The six adhesives evaluated were two epoxies, two polyesters, and two rubber base, one of the rubber base adhesives being supported on mylar. The test consisted of bonding Velcro fasteners to a purge bag, pressurizing the bag with helium, subjecting the pressurized bag to a blast of liquid nitrogen, and testing the attachment of the Velcro fastener under a load of 13 N. The only fasteners which remained attached under load were those bonded with the same epoxy adhesive used in the other applications.

CRYOGENIC PROPERTIES OF A POLYURETHANE ADHESIVE

Robbins, R. F. (National Bureau of Standards, Boulder, Colorado)
Polymers in Space Research (Proc., American Chemical Society Symposium,
Pasadena, Calif., July 15-17, 1968), 331-44, C. L. Segal, et al., Eds.,
Marcel Dekker, Inc., N.Y. (1970). Also in J. Macromol. Sci. Chem. A3,
No. 1367-80 (Nov 1969)

Several properties of a polyurethane adhesive were measured at cryogenic temperatures, demonstrating the test methods developed to measure the properties. Cast samples of the cured adhesive were used in all tests. Differential thermal analysis between 110 and 310 K was used to find low temperature transitions of the polyurethane. The glass transition was found at 235 K. Rebound resilience was measured with an instrument which automatically drops a ball onto the cooled sample, and detects the velocity with which the ball rebounds. Measurements between 110 and 380 K show a resilience minimum at 270 K, corresponding to the glass transition at 235 K, and relatively low resilience, indicating high energy absorption capability, at all lower temperatures. Standard tensile tests were conducted at 4, 76, and 195 K. Extreme plastic flow and high elongation were observed at 195 K. Only slight plastic deformation remained at 76 K, and at 4 K the adhesive showed decreased ultimate strength and brittle behavior. The tests showed that the polyurethane adhesive has high deformation and energy absorption capabilities well below the glass transition temperature. The experimental methods are applicable to performance evaluation in developing new materials.

Important references:

1. Robbins, R. F. and Reed, R. P., Advances in Cryogenic Engineering (K. D. Timmerhaus, ed.), 13, Plenum Press, NY (1968) p 252.

RTV SILICONES AS SEALANTS AND ADHESIVES FOR CRYOGENIC APPLICATIONS

Robinson, D. E., and Schreihans, F. A. (North American Aviation, Inc., Downey, Calif. Space and Information Systems Div.)

Materials Symposium (Proc., National SAMPE Symposium on Adhesives and Elastomers for Environmental Extremes, 7th, Los Angeles, Calif., May 20-22, 1964), Sect. 6, 1-29, Society of Aerospace Material and Process Engineers (1964)

A room-temperature vulcanizing (RTV) silicone rubber was evaluated for potential use as a sealant at temperatures as low as 20 K, for space vehicle applications. Initial screening led to the methyl-phenyl silicone RTV being selected for further evaluation. Low-temperature physical properties were found by tensile and compression testing of the cured elastomer. Two catalyst levels were compared, and a dimethyl silicone RTV was tested for comparison. Tensile strength, ultimate elongation, and tensile modulus data were obtained from 100 to 294 K. Compressive rebound data were derived from hysteresis loops with cyclic compressive loading over the same temperature range. Ultimate tensile strength and tensile modulus increased with decreasing temperature, while ultimate elongation and compressive rebound decreased with decreasing temperature. The properties of the dimethyl silicone changed at higher temperatures than did those of the methyl-phenyl silicone, indicating loss of flexibility near 210 and 170 K for the dimethyl silicone and methyl-phenyl silicone respectively.

The ultimate elongation of the methyl-phenyl silicone had unusual behavior, increasing with decreasing temperature to a peak near 170 K, then decreasing rapidly as temperature is further decreased. Less catalyst led to a higher peak in the elongation at slightly higher temperature. The methyl-phenyl silicone RTV was tested for compression set, shore hardness, and reversion (depolymerization) at temperatures above room temperature. Exposure to and aging at elevated temperature led to degradation of the material. The methyl-phenyl silicone was tested for seal leakage (fillet seals) at 20, 77, and 294 K, and for tensile-shear strength as an adhesive at 20 and 294 K. Seal leakage decreased as temperature was reduced to 77 K, but cracks developed and the seal leaked at sharp corners at 20 K. Elimination of sharp corners and application of vacuum before curing, to pull the elastomer into leakage paths, were found to produce good seals at 20 K. As an adhesive, the RTV silicone had about the same bond strength at 20 and 294 K, although the failures were cohesive at 294 K and adhesive at 20 K. The bond strengths were too low for structural applications, but useful for some non-structural bonds.

The authors conclude that the methyl-phenyl RTV silicone is useful from 165 to 475 K, and for 10 minutes or less up to 590 K. If high resiliency is not required, the low-temperature limit goes to 130 K. The elastomer has potential as a sealant below 130 K although additional work would be needed for large scale applications.

ADHESIVES FOR CRYOGENIC APPLICATIONS

Roseland, L. M. (Douglas Aircraft Co., Inc., Huntington Beach, Calif.) Cryogenic Properties of Polymers (Proc., NASA-Case Conference on the Properties of Polymers at Cryogenic Temperatures, Cleveland, Ohio, April 25-27, 1967), 17-28, T. T. Serafini, et al., Eds., Marcel Dekker, Inc., N.Y. (1968). Also in J. Macromol. Sci., Phys. B1, No. 4, 639-50 (Dec 1967)

This paper reviews some of the experience with cryogenic structural adhesives used in the saturn SIVB. The review pulls together material from several sources. Much of it is taken from a previous review (see abstract on page 112) which discussed materials and techniques known to improve joint strength in cryogenic applications. These included the use of tough polymer adhesives, the use of glass mat in the cured bond line, and the utilization of silanes as additives or primers to promote adhesion. Additional material was taken from a program to select the optimum fibrous material for modifying a polyurethane adhesive, reported in detail (see abstract on page 111). An anaerobic structural adhesive (a polyester) is described, which cures by catalytic action of metal ions when air is excluded. Lap shear strengths at 77, 297 and 394 K are shown.

Important references:

1. Saturn Data Summary Handbook, Douglas Missile and Space Systems Group Logistics Support Publication, Oct 1965.
2. Yurenka, S., Douglas Missile and Space Systems Group, Jan 1964. (Eng. Paper 1691).
3. Serman, S. and Toogood, J. B., Union Carbide Corporation, Silicone Division, Buffalo, NY.
4. Roseland, L. M., Douglas Missile and Space System Group, May 1967. (Eng. Paper 4191).
5. Loctite Corporation, Newington, Conn., June 1965. (Loctite Catalog 284).

EVALUATION OF ADHESIVES FOR POTENTIAL CRYOGENIC USAGE ON THE S-IVB

Roseland, L. M.

Douglas Aircraft Co., Inc., Santa Monica, Calif., Rept. No. SM-43086

(Apr 1963) 36 pp

A survey of commercially available structural adhesives was made to select systems having useful properties at cryogenic temperatures for possible future application on the Saturn S-IVB. Fifteen adhesives of two general types were evaluated. Room-temperature curing pastes included three modified epoxies, a flexibilized epoxy, a polyamide-epoxy, a modified nylon-filled epoxy, and a polyurethane. Heat curing films (maximum 450 K) included four nylon-epoxy unsupported films, a modified epoxy fabric film, a modified epoxy on nylon cloth, and two modified epoxy-phenolics on glass cloth.

Initial selection was based on tensile-shear and T-peel tests on aluminum-aluminum bonds at 20 and 394 K, and thermal shock in cycling between 219 and 344 K, plus button tensile tests at 20 and 394 K for the room-temperature curing pastes, and climbing drum peel tests at 20 K, RT, and 394 K for the 450 K cure adhesives. Three room-temperature curing pastes, the polyamide-epoxy, flexibilized epoxy, and polyurethane, were selected for further testing including tensile-shear, T-peel, sandwich tension, creep and fatigue. Three 450 K cure adhesives, a nylon-epoxy unsupported film, a modified epoxy-phenolic on glass cloth, and the modified epoxy fabric film, were selected for tests including tensile-shear, T-peel, climbing drum peel on glass or aluminum honeycomb core, creep, fatigue, and flexural shear on glass honeycomb core. On the basis of all tests, two adhesives, the modified epoxy fabric film and the polyurethane, were recommended for further evaluation with S-IVB liquid hydrogen vessels.

This report identifies the adhesives only by their commercial names. The report was rewritten containing the same data, but deleting all commercial names and identifying the adhesives only by type, and was presented at a SAMPE Symposium in 1964 (see citation in Secondary Documents).

EVALUATION OF FIBROUS MATERIALS IN A CRYOGENIC STRUCTURAL ADHESIVE

Roseland, L. M.

Douglas Aircraft Co., Inc., Santa Monica, Calif., Rept. No. SM-49202,
National Aeronautics and Space Administration Rept. No. NASA-CR-85121,
Contract No. NAS7-101 (Nov 1966) 34 pp

A three-phase test program was carried out to increase the reliability of adhesive bonding of attachments to the skin of the Saturn S-IVB. Previous work had indicated that embedding a glass fabric into the bond line improved the reliability. The adhesive was a two-part system, consisting of polyurethane adhesive base and diamine-curing agent, used in aluminum-aluminum bonds. Twenty-three different fibrous constructions were embedded in adhesive, and samples were tested for T-peel strength at 77 K. The five fabrics exhibiting highest strength included three of glass and two of dacron. These materials were tested for lap-shear strength of samples at 77 and 297 K, and the best three were selected for further tests. On the basis of T-peel and lap-shear tests from 77 to 355 K, a non-woven glass fiber mat was selected as the most promising of the reinforcements tested for cryogenic application.

This paper was partially rewritten and presented at a SPE Meeting in 1967 (see citation in Secondary Documents).

Important references:

1. Roseland, L. M., Douglas MP Report No. 2191, Catalog No. PDL 54783 (Jul 1965).
2. Roseland, L. M., Douglas SM Report No. SM-49146 (Aug 1965).

MATERIALS FOR CRYOGENIC USAGE

Roseland, L. M., Jr

Douglas Aircraft Co., Inc., Santa Monica, Calif., Missile and Space Systems Div., Paper No. 3579 (Oct 1965) 13 pp. Presented at Society of Plastics Industry, Reinforced Plastic Div. Conference, Chicago, Ill., February, 1966

A review of some methods for improving the properties of structural adhesives and composite materials at cryogenic temperatures is presented. The review discusses the use of tough polymers, the addition of adhesion promoting silanes, and the utilization of fibrous materials as fillers. Two adhesive systems, a polyurethane room-temperature curing paste adhesive and a 450 K curing nylon-epoxy film, are shown as examples of tough polymer adhesives, based on tensile-shear and T-peel results at 20 and 297 K. Results for the nylon-epoxy material are superior at room temperature, while the polyurethane has better properties at 20 K. Both materials showed little or no leakage of hydrogen through their cured bond lines after thermal shock tests between 20 and 273 K. The use of silane coupling agents is demonstrated by the use of a polyurethane adhesive with and without the addition of 1% or less of a silane, in bonds to aluminum and to glass, where the silane increased bond strengths at room temperature and 395 K without any decrease of strength at 20 K. The utilization of fibrous materials is demonstrated by the greatly decreased thermal contraction between 300 and 77 K of both epoxy and polyurethane resins, when modified by the addition of glass cloth. All three methods for improving strength were used in composites made with urethane resin with silane added and modified with glass fabric. T-peel strengths at room temperature more than doubled, while strengths at 77 K decreased but remained within acceptable limits.

This paper was rewritten and expanded somewhat, and presented as a conference paper (see abstract on page 109).

Important references:

1. Yurenka and Steven, Engineering Paper No. 1691, Douglas Missile and Space Systems Division (Jan 1964).
2. Douglas Laboratory Report MP 30,258 (Jul 1963) (Internal report).
3. Sterman, S. and Toogood, J. B., Union Carbide Corp., Silicone Division, Buffalo, New York.
4. Patten, P. M., Report No. SM 45975, Douglas Missile and Space Systems Division (Aug 1964).

THERMOELASTIC ANALYSIS OF SOLAR CELL ARRAYS AND THEIR MATERIAL PROPERTIES

Salama, M. A., Rowe, W. M., and Yasui, R. K.

Jet Propulsion Lab., Calif. Inst. of Tech., Pasadena, Rept. No. JPL-TM-33-626. National Aeronautics and Space Administration Rept. No. NASA-CR-135713, Contract No. NAS7-100 (Sep 1973) 112 pp

The thermoelastic properties of materials used in silicon solar cell arrays were determined, to help in predicting thermally induced stresses and failures in the arrays. RTV-type silicone rubber adhesives were considered to be the best materials for bonding solar cells to substrates and filter glasses to solar cells. The seven RTV silicone rubber adhesives tested include two dimethyl, three methyl-phenyl, and two methyl-vinyl(-phenyl) silicones. The thermal expansion coefficients between 77 and 470 K of six of these materials had been found in previous work, and were shown graphically in this report. Cured bulk specimens of the seven adhesives were tested at 89, 173, 248, 298, 373 and 473 K, both in tension and in compression. The results are displayed in the form of graphs of elastic moduli, ultimate strengths, and typical stress-strain curves. Poisson's ratios were determined between 100 and 470 K for the materials, and details of the photographic method developed for this measurement are included. Wide variations in the properties were observed, particularly at and below the transition temperatures. The phenyl-methyl silicones had more gradual and lower temperature transitions into brittle phases than the dimethyl silicones, and their behavior was considered more compatible with the behavior of the other materials used in the silicon solar cell array.

Important references:

1. Salama, A. M., Rowe, W. M., and Yasui, R. K., Conference Record of the Ninth IEEE Photovoltaic Specialists Conference, 146-157, Silver Spring, MD (May 1972).

DEVELOPMENT OF IMPROVED ADHESIVES FOR USE AT CRYOGENIC TEMPERATURES TO
-423°F

Sandler, S. R., Berg, F., and Kitazawa, G.

Borden Chemical Co., Philadelphia, Pa., Central Research Lab., Final
Summary Rept., July 13, 1963-August 31, 1965. National Aeronautics and
Space Administration Rept. No. NAS-CR-82602, Contract No. NAS8-11518
(Sep 1966) 79 pp

The program was carried out over a two-year period to develop new adhesives showing good tensile-shear and T-peel properties over the temperature range from 20 to 477 K. A large part of the effort went into evaluating polyurethanes and epoxy resins as cryogenic adhesives. Several other chemical structures were also investigated. The adhesives included a wide range of polyurethanes, copolymers of polyurethane with epoxies (which produced a new polymer system, poly-2-oxazolidones), polyisocyanurates, several bisphenol-A epoxies, a number of epoxy esters, and some modified epoxies which were flexibilized by the addition of polyether structures. The adherends used in most cases were 2014 aluminum, although some adhesives were tested on 321 stainless steel. The adhesives were evaluated by testing for tensile-shear strength at 77, 296, 394, and 477 K, with a few results at 4 K, and some tests of T-peel strength at 296 K.

Polyurethanes with a polyether backbone had the highest tensile-shear strengths at cryogenic temperatures, but had poor properties above room temperature. Modification of prepolymer structures and curing agents did not solve the problem. Raising cure temperature to 373 K for two hours gave satisfactory strengths up to 394 K for some systems. Copolymerizing the polyurethanes with epoxies having good high temperature properties produced systems with little improvement over the polyurethanes. Isocyanate-terminated polyurethane prepolymers were converted at 438 K to polyisocyanurates, which showed good tensile-shear strengths from 77 to 477 K, but attempts to lower the conversion temperature were unsuccessful. The epoxies could be formulated and cured at 373 K to have good tensile-shear strengths at temperatures from 77 to 477 K, but T-peel strengths at 296 K were very low. Best tensile-shear strengths at 77 K were found with epoxies having inferior high-temperature strength. Polyether structures were added to the epoxies in attempts to increase T-peel strength, but the high-temperature properties decreased as a result.

Thus, during the program, several adhesives were made which met the tensile-shear requirements over the entire temperature range, but none of them had satisfactory T-peel strength. The authors conclude that it is not feasible to produce an adhesive meeting all of the requirements,

since increased flexibility to meet T-peel requirements results in decreased high-temperature tensile-shear strength. Room-temperature curing of some polyurethanes and epoxies is sufficient where tensile-shear strength at 477 K is not required. Higher cure temperatures produce more cross-linking, resulting in better shear strength at elevated temperatures, but at the expense of lower T-peel strength.

Parts of this report have been published in other places. See papers by these authors listed in Secondary Documents.

Important references:

1. Hertz, J., Advances in Cryogenic Engineering 7, 336 (1962).
2. Smith, M. B. and Susman, S. E., Cryogenic Engineering 8, 300 (1963).
3. Kausen, R. C., Materials in Design Engineering (Sep 1964) pp 106.
4. Roseland, L. M., Seventh National Society of Aerospace Materials and Process Engineers Symposium on Adhesives and Elastomers for Environmental Extremes, May 20-22, 1964 at Los Angeles, California, Section 7.

CENUSIL -- A GOOD ACOUSTIC BOND FOR LOW TEMPERATURE ULTRASONIC MEASUREMENTS
Seidenkranz, T., and Hegenbarth, E. (Technische Hochschule, Dresden,
East Germany. Sektion Physik)
Cryogenics 15, No. 7, 422-3 (Jul 1975)

This note recommends a silicone rubber adhesive for low temperature acoustic bonds. Acoustic bonding requires less strength than structural bonding, the primary requirement being to transmit waves between a sample and a transducer. The silicone rubber recommended here cures at room temperature by means of humidity in the air. It was used to bond quartz transducers to molybdenum and to MgZn₂ samples, and transmitted longitudinal waves from 4.2 to 300 K and transverse waves up to 200 K. The bonds withstood cooling rates as high as 2 K/min and withstood frequent temperature cycling. This paper also lists a number of materials which have been used by various investigators for acoustic bonding at low temperatures. The materials listed include two stopcock greases, a silicone fluid, a Russian silicone, a mixture of gelatine and water, 3-phenyl propyl chloride, natural gas, and three metals. No data on these materials are given beyond general useful temperature ranges and references to the literature.

Important references:

1. Bateman, T. B., J. Acoust. Soc. Am. 41, 1011 (1967).
2. Bolef, D. I. and de Klerk, J., J. Appl. Phys. 33, 2311 (1962).
3. Featherston, F. H. and Neighbours, J. R., Phys. Rev. 130, 1324 (1963).
4. Dobbs, E. R., Hughes, E., Lawson, N. S., et al., J. Phys. E: Sci. Instr. 6, 309 (1973).
5. Matheson, A. J., J. Phys. E: Sci. Instr. 4, 796 (1971).
6. Strukov, V. B., and Shchegolev, J. F., Prib. i Tekh. Eksper. 5, 248 (1966).
7. Marshall, B. J., Phys. Rev. 121, 72 (1961).
8. Norwood, M. H. and Brisca, C. V., Phys. Rev. 112, 45 (1958).

GLUE COMPOSITIONS FOR CRYOGENIC DEVICES

Shapovalenko, V. V., Goldshtein, E. I., Pomoshnikova, M. V., and Voitenko, L. I. (Academy of Sciences of the Ukrainian SSR, Kharkov. Physicotechnical Institute of Low Temperatures)

Instrum. Exp. Tech. 17, No. 3, Pt. 2, 903-4 (May-Jun 1974) Transl. of Prib. Tekh. Eksp., No. 3, 233-4 (May-Jun 1974)

Several adhesive formulations are recommended for use at cryogenic temperatures in bonding materials with widely differing thermal expansion coefficients. Twelve formulations are shown, made up of various combinations of three resins, three curing agents, a plasticizer, and four fillers. The formulations are said to be characterized by relatively large content of curing agent, for improved elasticity, and small content of filler, for improved adhesive bonding to surfaces. Except for the fillers, the components are identified by their Russian designations. One of the resins is identified as an epoxy-silicone. Pot lives of the formulations range from one hour to several months. Application temperatures and curing cycles are given. The adhesive formulations were used successfully to bond various metals to glasses and to germanium. Bonds were tested for leak-tightness in superfluid helium, and withstood repeated thermal cycling.

Important references:

1. Anashkin, O. P., Pribory i Tekh. Eksperim., No. 2, 225 (1972).

DEVELOPMENT OF LIQUID OXYGEN COMPATIBLE ADHESIVE SYSTEM

Shdo, J. G., and Hoggatt, J. T.

Boeing Co., Seattle, Wash. Final Rept., Part II. National Aeronautics and Space Administration Rept. No. NASA-CR-72502, Contract No. NAS3-7952 (Dec 1968) 54 pp

Candidate adhesive systems were evaluated for LOX compatibility and adhesive characteristics, for application in fabricating light-weight expulsion bladders for liquid oxygen systems. A polyimide film was selected as the bladder material. Although its impact-sensitivity in liquid oxygen was marginal, it was less impact-sensitive than other polymeric film materials. The adhesive used to fabricate bladders was required to be less impact-sensitive than the polyimide film, to prevent decreasing the already-marginal LOX compatibility.

The nine adhesives evaluated were a copolymer of vinylidene fluoride and chlorotrifluoroethylene, a copolymer of perfluoropropylene and vinylidene fluoride, a one-component fluoroelastomer LOX-compatible coating material used in the Apollo space capsule, a two-component room-temperature-curing version of the coating material, a copolymer of perfluoropropylene and tetrafluoroethylene used as a hot-melt adhesive film, and carboxyl-nitroso rubber elastomers in the forms of a coating solution, a liquid, a pressure sensitive adhesive, and a gum-stock. The screening tests consisted of tensile-shear strengths of polyimide film-polyimide film bonds at 77 and 297 K, LOX impact tests of bonds and of pure adhesives, and twist-flex tests of bonds at 77 K. The copolymer of vinylidene fluoride and chlorotrifluoroethylene was selected as the best adhesive for the application, with the carboxyl-nitroso rubber coating solution next in performance. Chemical modification of the polyimide film surface was found unnecessary for adhesion. The best adhesive was used to fabricate a single-ply bladder, which was tested for integrity after ten expulsion cycles at 77 K. The bladder was sectioned and used in LOX ballistic impact tests and lap-shear tests of seam areas. The fabricated bladder and the seam areas successfully met the program objectives.

Important references:

1. Hoggatt, J. T., NASA CR-72418, The Boeing Company, Seattle, Washington (June 1968).
2. Lank, R. F., NASA TMX-15555, Lewis Research Center, Cleveland, Ohio (Apr 1968).
3. Testing Compatibility of Materials for Liquid Oxygen, NASA MSFS-SPEC-106B.

Important references (continued):

4. Key, C. F. and Riehl, A., NASA TMX-895 (Aug 1964).
5. Hoggatt, J. T., NASA CR 72134, The Boeing Company, Seattle, Washington (Jan 1967).
6. Hoggatt, J. T., The Boeing Company, Document D2-237781-1, Seattle, Washington (Jan 1965).
7. Pope, D. H. and Penner, J. E., NASA CR-72115, Beech Aircraft Corporation, Boulder, Colorado (Mar 1967).

SPECIFIC HEAT OF BF-4 CEMENT IN TEMPERATURE RANGE 20-360 DEGREES K
Sklyankin, A. A.
Instrum. Exp. Tech., No. 4, 808-9 (Feb 1962). Transl. of Prib. Tekh.
Eksp., No. 4, 180 (Jul-Aug 1961)

This note reports results of measurements of the specific heat of a Russian adhesive. The material retains its mechanical strength and electrical insulation properties from cryogenic temperatures to above room temperature, and is often used in constructing equipment for low-temperature experiments. The adhesive is identified only by its Russian designation. It was applied in several layers to a copper base, specific heats were measured from 20 to 360 K, and specific heat of the copper was subtracted from the total. Results are presented in tabulated and graphical forms. The specific heat increased nearly linearly with temperature up to 310 K, then showed a more rapid increase up to 345 K. The anomalous behavior between 310 and 345 K was probably caused by phase transformation associated with softening of the adhesive in this temperature range.

Important references:

1. Kalinkina, I. N., PTE No. 1, 146 (1958).

INVESTIGATION OF COMBINED EFFECTS OF RADIATION AND VACUUM AND OF RADIATION AND CRYOTEMPERATURES ON ENGINEERING MATERIALS. VOLUME II: RADIATION-CRYOTEMPERATURE TESTS

Smith, E. T.

General Dynamics/Fort Worth, Tex., Annual Rept. No. FZK-161-2, National Aeronautics and Space Administration Rept. No. NASA-CR-51140, Contract No. NAS8-2450 (Nov 1962) 253 pp

Previous work in the field of radiation effects has shown that drastic changes can be induced in the engineering properties of non-metallic materials as a result of exposure to ionizing radiation. The materials tested in this program included adhesives, seals, thermal insulation, electrical insulation, structural laminates and thermal control coatings. Radiation exposure took place at 20 K, 77 K and 298 K at three different doses. Following this, measurements were made of shear-tensile strength, ultimate tensile strength, ultimate elongation, stress-strain, leakage, compression-deflection and spectral reflectivity. Each material was tested in each of nine different conditions of temperature and radiation dose. In the case of irradiation at low temperature and subsequent low temperature test, the sample was not warmed so that radiation induced defects would not be annealed out.

The adhesives investigated were a polyurethane and an unsupported epoxy-mylar tape. The adhesives were tested only in the lap-shear mode. The polyurethane demonstrated increased shear-tensile strength after irradiation at room temperature. At cryogenic temperatures, the strength before irradiation was considerably higher than the room temperature value. Radiation then served to reduce the strength somewhat, but the value remained higher than the room temperature/no irradiation level. The author recommends this adhesive highly for use in the combination of environments tested (up to a dose level of 5×10^{10} ergs/gm of gamma radiation). The epoxy-mylar tape adhesive suffered severe degradation in the shear-tensile tests at all temperatures following irradiation at a dose level of 3×10^{10} ergs/gm and is not recommended for use in this kind of environment.

MEASUREMENT OF THE COMBINED EFFECTS OF NUCLEAR RADIATION AND CRYOTEMPERATURES ON NON-METALLIC SPACECRAFT MATERIALS

Smith, E. T.

General Dynamics/Fort Worth, Tex., Rept. No. FZM-2937 (1963) 36 pp.

Presented at AIEE Summer General Meeting, Toronto, Canada (Jun 17-21, 1963) Paper No. CP 63-1175

This paper presents the experimental results of a program to measure the combined effects of nuclear radiation and cryogenic temperatures on the mechanical (tensile and compressive) properties of nonmetallic materials. The purpose of the program was to aid in the selection of nonmetallic structural materials for use in nuclear-powered spacecraft. The materials tested included two adhesives, two mechanical seal materials, two thermal insulation materials, two electrical insulation materials and a structural laminate. The materials were tested at ambient conditions with no irradiation and at 20 K and 77 K at zero radiation and up to 6×10^{10} ergs/gm. The irradiation and subsequent testing were done without warming the sample, so that no chance was given for annealing out the irradiation induced defects.

The adhesives were tested in the tensile-shear mode. The adhesives tested were a polyurethane and a nylon-epoxy. The polyurethane adhesive exhibited increasing strength with decreasing temperature (strength nearly tripled between ambient and 20 K). Irradiation at ambient temperature increased strength, while it decreased the strength at both 20 K and 77 K. The degradation caused by radiation at cryogenic temperatures was not severe and the strength remained higher than the corresponding ambient values. No quantitative results are given for the nylon-epoxy adhesive, but the author reports severe radiation degradation and a lowering of strength with temperature at all radiation levels. The nylon-epoxy is not recommended for use while the polyurethane is recommended at all temperatures below ambient and to radiation levels of 5×10^{10} ergs/gm. Most of the results of this paper are used by different authors in reference abstracted on page 83.

Important references:

1. Kerlin, E. E. and Smith, E. T., General Dynamics/Fort Worth Report FZK-152, 15 Aug 1962.
2. Smith, E. T., General Dynamics/Fort Worth Report FZK-161-2, 5 Jan 1963.

DEVELOPMENT OF ADHESIVES FOR VERY LOW-TEMPERATURE APPLICATION

Smith, M. B., and Susman, S. E. (Telecomputing Corp., San Diego, Calif. Narmco Research and Development)

Adv. Cryog. Eng. 8 (Proc., Cryogenic Engineering Conference, Los Angeles, Calif., August 14-16, 1962), 300-5, K. D. Timmerhaus, Ed., Plenum Press, N.Y. (1963)

The purpose of the program reported in this paper was the development or selection of an adhesive for bonding hardware to the skin portion of cryogenic tanks for spacecraft. The adhesive should also be suitable when in contact with the cryogenics. The surface preparation required should be simple with curing at ambient conditions. Tests included shear-tensile and tee-peel strength. Adherends were 7075 aluminum alloy and 17-7PH stainless steel with the tests conducted at 20 K, 77 K, 273 K, 325 K and 366 K. Nylon-epoxy adhesives gave the best results, but required 2 atmospheres pressure for curing and were thus not suitable as currently available. Various kinds of surface preparation were tested including solvent degrease, sandblast, acid etch, anodizing, and hydrogen peroxide etch. Sandblast and anodizing proved best. Fillers were studied with respect to the epoxy-polyamide system. Fillers included metallic and polymeric materials. A powdered nylon filler was selected for further study. The filler approach was expanded to include substrate films such as halogenated polymers, nylon, mylar, etc. Teflon FEP contributed the most to peel and tensile shear strengths.

Polyurethane adhesives were included in the study even though commercial systems were not at that time available. Formulations were developed during the project which had tensile shear strengths comparable to epoxy-nylon systems. The three systems tested, nylon-epoxy, epoxy polyamide and polyurethane, showed promise, but none fulfilled all of the objectives of the project. The greatest shortcoming of all systems was high temperature strength. A comment in the discussion states that all have been impact tested in liquid oxygen and were found to be non-compatible with LOX.

DEVELOPMENT OF ADHESIVES FOR VERY LOW TEMPERATURE APPLICATION

Smith, M. B., and Susman, S. E.

Narmco Research and Development, San Diego, Calif., Final Summary Rept.
National Aeronautics and Space Administration Rept. No. NASA-CR-52879,
Contract No. NAS8-1565 (May 1963) 216 pp

The work reported here was sponsored by NASA and was directed toward evaluating and testing adhesives and adhesive systems for bonding hardware to cryogenic tanks on spacecraft. The work consisted of a literature survey and comparison of available data. This was followed by a three step experimental selection and screening process.

- Step 1. Shear-tensile and tee-peel tests at 77 K and 298 K.
- Step 2. Shear-tensile and tee-peel tests from 20 K to 400 K.
- Step 3. The most promising adhesives of each type were further tested by means of mechanical shock, butt-tensile, compression and liquid oxygen compatibility tests.

The classes of adhesives tested were nylon-epoxy, epoxy-polyamide, polyurethane and fluorocarbon film systems. The external effects studied included filler material, film supporting media and surface preparation. The adherends included a wide variety of aluminum alloys and stainless steels. Six adhesives were selected as a result of the program. These adhesives were a nylon powder filled epoxy-polyamide, a teflon FEP film epoxy-polyamide composite, two polyurethanes, a glass fabric supported polyurethane and a teflon FEP hot-melt adhesive. All except the last system were cured at ambient temperature and contact pressure. All of the six adhesives provide excellent, moderate, low and very low temperature strength, excellent toughness at low and very low temperatures and superior peel strength at low temperatures. Only the Teflon-FEP hot-melt adhesive was found to be compatible with liquid oxygen. Other factors tested included cure time, thermal cycling (77 K to 298 K), glue-line thickness and thickness mismatch. The report also includes a complete description of each developed adhesive including surface preparation and optimum curing. This is a later and more complete version of the paper abstracted on page 123.

Important references:

1. Advances in Cryogenic Engineering 6, 627.
2. Miller, R. N., et al., paper presented at the Washington Meeting of American Chemical Society, Div. of Organic Coatings and Plastics Chemistry 22, No. 1 (1962).
3. Hauser, R. L. and Rumpel, W. F., Martin Co., Denver, Colorado, Paper No. 82, Cryogenic Engineering Conference, Los Angeles, California (1962).

THE THERMAL PROPERTIES OF SAMPLE ADDENDA USED IN $T < 1$ K SPECIFIC HEAT MEASUREMENTS: 1 - SPECIFIC HEAT AND THERMAL CONDUCTIVITY OF GENERAL ELECTRIC 7031 VARNISH

Stephens, R. B. (Cornell Univ., Ithaca, N.Y. Physics Dept.)
Cryogenics 15, No. 7, 420-2 (Jul 1975)

The thermal conductivity and specific heat of a varnish were measured below 1 K. The varnish, a polyvinyl phenolic, is widely used as an insulating adhesive in low-temperature work, particularly to attach heater wires and thermometers to calorimetric samples. Other measurements of specific heat and thermal conductivity of the material had been made previously, but at temperatures above 1 K, while the varnish is used at temperatures down to 0.05 K. A bulk sample was cast and air dried very slowly over fourteen months to produce a homogeneous, bubble-free test specimen. Specific heats were measured from 0.219 to 1.53 K, and thermal conductivities from 0.0694 to 0.994 K. Results are shown in tabulated and graphical forms. The results of previous measurements are shown on the same graphs for comparison, and substantial systematic differences appear in the data. The author concludes that such differences are to be expected because of the nature of the adhesive and probable differences in residual solvent content with different sample preparations. The variations make the data inaccurate for use in addenda corrections in low-temperature calorimetry, but estimates are possible.

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1. Heessels, J. T., Cryogenics 11, 483 (1971).
2. McTaggart, J. H. and Slack, G. A., Cryogenics 9, 384 (1969).
3. Cude, J. L. and Finegold, L., Cryogenics 11, 394 (1971).
4. Phillips, N. E., Phys. Rev. 114, 676 (1959).

A PRELIMINARY EVALUATION OF SILANE COUPLING AGENTS AS PRIMERS AND ADDITIVES
IN POLYURETHANE BONDING PROCEDURES

Thompson, L. M., and Hill, W. E.

National Aeronautics and Space Administration, Marshall Space Flight
Center, Huntsville, Ala., Tech. Memo. No. NASA-TMX-53676 (1967) 15 pp

Polyoxytetramethylene based polyurethane adhesives have proved to possess high strength at cryogenic temperatures. The strength of this class of bonding agents decreases rapidly at ambient temperatures and above. In addition these adhesive formulations often exhibit large scatter in a given set of lap shear tensile and T-peel tests. This publication presents results of attempts to improve the higher temperature utility and overall reliability of the polyurethane adhesives by application of silane coupling agents to the adherends in a priming technique or by addition of silane coupling agents directly to the polyurethane resin system. Three commercially-available silane derivatives were evaluated as primers for cleaned aluminum adherends. Both tensile-shear and T-peel tests were conducted on the three different primer configurations and the one case where the silane was added to the resin system, and all were compared with unprimed controls. The tests were conducted at 91 K, 298 K and 367 K. In all four cases, increased bond strengths were obtained. These increases occurred at all three temperatures, with the improvement at 367 K the most pronounced. The latter was also the most sought after. The improvement at 367 K compared with the control was about three times for tensile-shear and twice for T-peel.

STRESS-STRAIN BEHAVIOR OF ADHESIVES IN A LAP JOINT CONFIGURATION AT AMBIENT AND CRYOGENIC TEMPERATURES

Tiezzi, G. J., and Doyle, H. M. (McDonnell-Douglas Corp., Santa Monica, Calif.)

Polymers in Space Research (Proc., American Chemical Society Symposium, Pasadena, Calif., July 15-17, 1968), 295-317, C. L. Segal, et al., Eds., Marcel Dekker, Inc., N. Y. (1970). Also in J. Macromol. Sci., Chem. A3, No. 7, 1331-53 (Nov 1969)

Certain applications of adhesives require a knowledge of the stress distribution in the glueline of lap joints which in turn necessitates knowledge of the shear modulus of the adhesive system. Previous work indicates that the shear modulus cannot be accurately predicted from shear-tensile tests of lap specimens. The purpose of the experimental program reported here is the development of specimen configurations, test equipment and test methods for determining the shear modulus of adhesives in the general range of 77 K to 300 K. The specimen configuration selected employs a double lap joint with four adhesive layers (two in parallel and two in series), with the adherends composed of aluminum strips. Stress-strain distributions were determined by two methods - strain gage and photostress coatings. Both methods gave similar results, except that the photostress method is not suitable for cryogenic temperatures.

Room temperature results are given for commercially-available epoxy and polyurethane adhesives as well as 77 K results for the polyurethane system. These results are compared with values predicted using a theoretical model fitted to a polynomial expansion. The agreement is excellent.

The authors feel that they have developed a test method which models very closely the performance of the adhesive systems in actual practice.

CRYOGENIC/HIGH TEMPERATURE STRUCTURAL ADHESIVES

Vaughan, R. W., and Sheppard, C. H.

TRW Systems Group, Redondo Beach, Calif., Final Rept., National Aeronautics and Space Administration Rept. No. NASA-CR-134465, Contract No. NAS3-16780 (Jan 1974) 69 pp

The objective of the program was to develop a cryogenic-high temperature structural adhesive system for application to the space shuttle. The primary requirement was for useful properties at temperatures from 20 to 589 K. A literature survey identified nine potential candidate materials, four polyimides, a polyquinoxaline, two polyphenylquinoxalines, a polybenzimidazole, and a polybenzothiazole. The processing requirements for bonding with the polyquinoxaline, the polybenzimidazole, and the polybenzothiazole were too severe and not conducive to production techniques, and these materials were not evaluated further.

Three polyimides and a polyphenylquinoxaline were selected for evaluation, and the remaining polyimide and polyphenylquinoxaline were considered similar to those being evaluated. One polyimide had been used to bond titanium. A new formulation including an antioxidant was developed for use with stainless steel. Another polyimide which had been used to make glass composites proved to be a poor adhesive for metals, and was eliminated from the program. Detailed properties tests were made on the two polyimides (one of them having two variations) and the polyphenylquinoxaline, with Ti-6Al-4V titanium alloy and 17-7 PH stainless steel as adherends. Tests included tensile-shear strengths at 20, 52, 295, 533, and 589 K; thermal shock between 20 and 589 K; stressed and unstressed thermal aging at 477, 533, and 589 K; and thermal expansion coefficients between 20 and 589 K. There was insufficient polyphenylquinoxaline for complete evaluation, and testing on this material did not include cryogenic temperatures. The high-temperature results indicated high enough promise to warrant further developmental studies of the adhesive.

Both of the polyimides provided sound structural joints with titanium alloy or stainless steel at all temperatures between 20 and 589 K, with the two systems providing approximately equivalent properties. The authors prefer one polyimide over the other on the basis of easy processing and better bondlines, particularly in large surface area joints. A short version of this report was presented as a paper at a SAMPE Symposium (see citation in Secondary Documents).

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THERMAL CONTACT AND INSULATION BELOW 1 DEGREES K

Wheatley, J. C., Griffing, D. F., and Estle, T. L. (Illinois Univ., Urbana)

Rev. Sci. Instrum. 27, No. 12, 1070-7 (Dec 1956)

Heat transfer across adhesive bonds was measured below 1 K. The investigation was concerned with the problem of making thermal contact between a single crystal of a paramagnetic salt and other materials. When heat is conducted through a thermal link with low thermal resistance, such as crystalline quartz or high purity copper, most of the thermal resistance occurs at the interface between the crystal and the thermal link, and the thermal resistance of a bonding agent is an important consideration. The adhesive considered was a varnish diluted with toluene. It was used to bond crystals of ferric ammonium alum to opposite ends of thermal links, which have negligible thermal resistance. One type of thermal link was a single crystal of quartz, and the other type was a cylinder of unannealed 99.999% copper. Heat flow from one paramagnetic crystal to the other, by way of two adhesive bonds and a thermal link, was measured. Results are presented in the form of constants in a heat flow equation. Results are also given for a frozen vacuum oil used as the bonding agent with a quartz thermal link. The temperature range for all data was 0.16 to 0.27 K. The data helped in establishing guiding principles for the design of sample holders with reproducibly low heat leaks at temperatures below 1 K.

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THE STRENGTH OF ADHESIVE BONDS INVOLVING METALS WITH ORGANIC AND CERAMIC ADHESIVES AT LOW AND HIGH TEMPERATURES (DIE FESTIGKEIT VON METALLKLEBVERBINDUNGEN MIT ORGANISCHEN UND KERAMISCHEN KLEBERN UNTER TIEFEN UND HOHEN TEMPERATUREN)

Witt, W. (Hanover Technische Hochschule, West Germany)

Metallkleben--Grundlagen, Technologie, Prufung, Verhalten, Berechnung, Anwendung, A. Matting, Ed., 332-45, Springer-Verlag, Berlin, Germany (1969)

The results reported in this paper are summarized from other sources and represent mechanical property testing of lap-shear tensile specimens in three temperature regions: low temperatures (20 K - 300 K), moderate temperatures (220 K - 600 K) and high temperatures (300 K - 1100 K). Polymeric adhesives are used for the first two regions and ceramic adhesives for the last. The materials are to be used for joining construction metals such as steel. The low temperature properties are for epoxy-nylon, epoxy-phenolic, and phenolic-nitrile adhesives, as well as qualitative discussion of polyurethane and alumina-filled epoxy adhesives. Commercially-available adhesives are listed for the various formulations. The strength test results for the cryogenic adhesives are such as to inhibit their use when exposed to temperatures much above room temperature. All of the test results shown in the paper stop at 300 K so that the downward trend is not very apparent in the graphical data.

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PPO FOAM INTERNAL INSULATION

Yates, G. B., and Tatro, R. E. (General Dynamics Corp., San Diego, Calif.)

Space Transportation System Propulsion Technology Conf., Vol IV. Cryogens. National Aeronautics and Space Administration, Marshall Space Flight Center, Huntsville, Ala., 1439-52 (Apr 6-7, 1971)

An open-cell polyphenylene oxide foam insulation was developed for use as internal insulation for space shuttle liquid hydrogen tanks. Three adhesives, two epoxies and a polyurethane, were tested for bonding the PPO foam to aluminum. PPO-aluminum bonded samples were tested for face tensile, compressive, core shear, and climbing-drum peel strengths at temperatures from 20 to 422 K. In all cases, failure was in the foam, so that test results were foam strengths rather than adhesive strengths. Tensile-shear tests resulted in selection of one epoxy and the polyurethane adhesive for bonding. Tensile-shear strengths of these two materials were reported at 20 K and 422 K. PPO-aluminum specimens bonded with the polyurethane adhesive were tested for structural integrity through 400 load cycles at 20, 294, and 394 K, with no observable failures, cracks, or deterioration in any sample. The conclusions drawn from the testing were that the adhesives were compatible with PPO foam and bond strengths were sufficient for the intended use over the temperature range tested.

DEVELOPMENT OF ORGANIC SEALANTS FOR APPLICATIONS AT VERY LOW TEMPERATURES

Zelman, I. M., Akawie, R. I., and Bahun, C. J.

Hughes Aircraft Co., Culver City, Calif., Rept. No. P 62-29, National Aeronautics and Space Administration Rept. No. NASA-CR-51727, Contract No. NAS8-2428 (Jun 1962) 79 pp

The purpose of this program was to develop sealants and seal materials for spacecraft use at very low temperatures (down to 20 K). The approach taken was a literature survey, followed by screening of polymer systems having the lowest possible glass transition temperature and the least tendency for crystallization, and then modification of these compounds to achieve better low temperature properties. The candidate sealants tested included various proprietary polymers, polysulfides, epoxies, silicones, polybutadienes, polyurethane, epoxy-silicones, and fluorosilicones.

The first screening test was a flexibility test at 76 K wherein the candidate material is cured on an aluminum strip and bent over a 5 cm radius mandrel. All candidates exhibited brittle failure, however two polyurethane base sealants and two silicone base sealants showed the most low temperature flexibility and were used for modifications. The modifications consisted of compounding with a variety of fillers and curing agents. The purpose of the modifications was to increase the low temperature bend strength and to reduce the thermal contraction to match the metal substrate. Small particles of glass fibers were found to be reasonably successful in accomplishing both purposes.

One specimen was tested in five vibration modes at 76 K and up to 50 g's with good results. The program also included a configuration test wherein cracks in simulated tanks were sealed and thermal cycled between 76 and 296 K. Polyurethane sealants with glass fiber filler (30 parts by weight) did not fail on repeated immersions in liquid nitrogen.

Further work must be done on incorporating the glass fibers in the resin. Work was also continuing on the synthesis of new silicone polymers.

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